OPTIMISING RESIN CONSUMPTION, PRESSING TIME AND DENSITY OF PARTICLEBOARD MADE OF MIXES OF HARDWOOD SAWMILL RESIDUE AND CUSTOM FLAKED SOFTWOOD

A thesis submitted in fulfillment of the requirements of the degree of

DOCTOR OF PHILOSOPHY

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DECLARATION

I declare that content of this thesis is the result of my own work, except where the acknowledgement has been made. The work has not been submitted or published previously. The content of the thesis is the result of work which had been carried out since the commencement of my PhD.

Kee Kong Wong

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ABSTRACT

Shortages of raw materials, constraints on natural resources and concerns about the carbon footprint of products have created a significant interest in environmentally-friendly or 'green' building materials, and the particleboard industry, which utilizes custom-flaked softwood as the major raw material, has been seeking alternative raw materials. Hardwood sawmill waste, which is available in excess and currently disposed of by burning, has been explored in this thesis as a part-replacement for particleboard manufacture. Earlier attempts at RMIT University to make particleboard in the laboratory from 100 % sawmill waste revealed that, whilst a product which satisfies the performance requirements of general purpose particleboard can be produced, higher resin consumption, longer pressing time and a higher board density could inhibit acceptance by the industry. The work presented here is aimed at producing an optimized particleboard product with a mix of softwood and hardwood residue to overcome the constraints identified earlier. The major hypothesis of the work was that boards made with mixes of hardwood and softwood would have better compaction and hence improved mechanical properties.

In order to establish the directions for the research, work commenced with a feasibility analysis of the economic and environmental benefits of using hardwood sawmill waste as a raw material for particleboard production. This was followed by a comprehensive literature review to identify the variables to be explored, the methodology of optimization of board properties and current industry practice. The theory of Design of Experiments (DOE) was selected as a possible method of identifying significant influencing parameters and later developing a process model. The procedure for making three-layer particleboard in the laboratory was established after pilot trials.

Four series of experiments were carried out during the research project. The first series was designed as a screening program to identify significant variables affecting particleboard. Press temperature and pressing time, hardwood proportion in the surface, moisture surface and moisture core were established as significant variables. However, the outcome of this first series of experiments indicated that mixtures of hardwood and softwood will not lead to any improvement in board properties. In the second series of experiments, the range of variables was adjusted to optimize the board properties, based on the outcomes of the first

series and only hardwood residue was used in the surface layers. The outcomes indicated that the acidity of the hardwood particles as well as the strength of the hardwood flakes affect the board properties and the hardwood-softwood configuration should be carefully controlled to manage the board properties. A third series of experiments was designed to explore the best configuration of hardwood-softwood in the boards. This work resulted in identifying that hardwood in the core and softwood in the surface would be the best configuration for optimum board properties. It was found that hardwood particles are not suitable in the surface layers as the acidic property of hardwood might accelerate the UF resin curing, leading to uneven curing throughout the board. This results in over-curing of the board. The best configuration of hardwood and softwood throughout the board was found to be softwood-only surface with hardwood-only core. After obtaining the most suitable hardwood/softwood configuration throughout the board, a final series of experiments was then carried out to establish the best processing parameters for board with satisfactory properties. Process models were then generated to predict board properties for board optimization. Using the model, the processing parameters and material parameters for an optimized board were identified. This board was then manufactured and the results used to validate the process model.

In order to understand the mechanisms underlying the observed behaviour, further investigations were conducted on resin curing of the boards. The board surfaces and the thermal analysis of different hardwood and softwood composites were analysed. The General Area Diffraction Detector System (GADDS) was used for the board surface studies while Thermogravimetric Analysis (TGA) was utilised to study the thermal behaviour of the hardwood and softwood composites. The results established a better understanding of the de-composition of hardwood and softwood composites. Based on knowledge of the de-composition temperature of both raw materials, the highest pressing temperature which can be adopted can be established.

LIST OF PUBLICATIONS DURING THE PhD CANDIDATURE

1. K. K. Wong, S. Setunge and M. Jollands 2009 ‘Economic and environmental benefits of using hardwood sawmill waste as a raw material for particleboard production’, Journal of Earth and Environmental Science - Water, Air and Soil Pollution, Vol. 9, no. 5 – 6, pp. 485 – 494

2. K. K. Wong, S. Setunge and M. Jollands 2010 ‘Particleboard production using mixtures of softwood and hardwood residues’, 21st Australian Conference on the Mechanics of Structure and Materials, Melbourne Australia. CRC Press 2010, pp. 573 – 578

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ABBREVIATIONS

ΔS	Increment of Deflection at the Mid-length of the Test Piece, corresponding to ΔW
ΔW	Increment in Load
A	Effect of Factor A (Chapter 4)
A	area of board (Chapter 5)
A	Surface Moisture Content (Chapter 6)
a,b	Length and Width of The Test Piece
$a_0...a_m$	The regression coefficient
AB	Effect of combination Factor A and B (Chapter 4)
ANOVA	Analysis of Variance
APFC	Asia-Pacific Forestry Commission
AS/NZS	Australian and New Zealand Standard
B	Effect of Factor B (Chapter 4)
b	Width of the Test Piece (Chapter 5)
B	Core Moisture Content (Chapter 6)
BS EN	British Standard
C	Effect of Factor C (Chapter 4)
C	Surface Resin Content (Chapter 6)
CCO_2	Specific Mass Carbon Dioxide
C*f*	Specific Carbon Content in the fuel
C*m*	Specific Mass Carbon
CO_2	Carbon dioxide
Cu-Kα	Radiation

D	Core Resin Content (Chapter 6)
DOE	Design of Experiments
DSC	Differential Scanning Calorimetry
DTG	Derivative of thermo gravimetric
E	Hardwood portion in Core (Chapter 6)
E	The Error term (Chapter 9)
f	Tensile Strength Perpendicular to plane of the Panel of Each Test Piece (Chapter 5)
F	Pressing Time (Chapter 6)
FAO	Food and Agriculture Organization
Fmax	Breaking Load
FTIR	Fourier Transform Infrared Spectroscopy
G	Pressing Temperature (Chapter 6)
GADDS	General Area Diffraction Detector System
$H_2C_2O_4$	Oxalic Acid
H_2SO_4	Sulphuric Acid
HCI	Hydrochloric Acid
h*f*	Specific Energy Content
HMR	High Moisture Resistance
I	Coefficients for the calculation of the constant effect (Chapter 4)
IB	Internal Bonding
ICDD	International Centre for Diffraction Data
JIS	Japanese Industrial Standard
k	Number of parameters (Chapter 4)
kPa	Kilo Pascals

kV	Kilovolt
L	Distance between the Centres of the supports
LSD	Least Significant Difference
m	Mass of Test Pieces
mA	mega Amphere
MC	Moisture core
MDF	Medium density fibreboard
MDI	Methylene diphenyl disocyanate
MF	Melamine formaldehyde
MJ	Megajoule
MOE	Modulus of Elasticity
MOR	Modulus of Rupture
MPa	Mega Pascal
MS	Moisture surface
MUF	Melamine Urea Formaldehyde
n	Number of Observations made
NH_4Cl	Ammonium Chloride
$NH_4H_2P0_4$	Ammonium dihydrogen phosphate
NIST	National Institute of Standards and Technology
P	Probability of Null Hypothesis (Chapter 2)
ρ	Density
p	Number of Model Parameters (page 60)
PF	Phenol-formaldehyde
qCO_2	Specific CO_2 Emission

R&D	Research and development
R^2	Regression coefficient
R^2_{adj}	Adjusted Regression coefficient
RC	Resin core
rpm	Round per minute
RS	Resin surface
S-H	Softwood only in surface and hardwood only in core
SHC	Screw Holding Capacity
SSE	Sum of squares of error component
SST	Sum of squares of test statistical values
STA	Simultaneous Thermal Analysis
t	Thickness of Board (Chapter 5)
t	Thickness of the Test Piece (Chapter 5)
T	Test Statistical Value
TG	Thermo gravimetric
TGA	Thermogravimetric Analysis
TS	Thickness Swelling
UF	Urea-formaldehyde
V	Resolution (Chapter 4)
V	Volume of Board (Chapter 5)
v	Volume of Test Piece (Chapter 5)
W	Ultimate Failure Load
WA	Water Absorption
W_{core}	Weight of Core

W_{fine}	Weight of Fine Particles
Wh	Measured Weight of Hardener
$W_{h(solid)}$	The Solid Weight of Hardener
$W_{h(water)}$	The Water Weight of Hardener
$W_{hardwood}$	Measured Weight of Hardwood
W_{mulch}	Weight of Mulch Particles
W_p	Measured Weight of Wood Residues
$W_{p(measured)}$	Measured Weight of Dry Wood Residues
$W_{p(solid)}$	The Solid Weight of Wood Residues
W_r	Measured Weight of Resin
$W_{r(solid)}$	The Solid Weight of Resin
$W_{r(water)}$	The Water Weight of Resin
$W_{softwood}$	Measured Weight of Softwood
W_{total}	Total Weight of Board
$W_{total(water)}$	The Total Weight of Water
$W_{w(added)}$	The amount of Water Added
$x_1 ... x_m$	The Factors Being Studied
XRD	X-ray diffraction
Y	Selected Response
ρ_{target}	Target Board Density
σ^2	Variance
χ_d	crystallinity
S_a	armophous integrated area

S_{cr} sum of the area of crystalline peak

CrI degree of crystallinity

I_{am} maximum intensity of the amorphous peak

Chapter 1

INTRODUCTION

1.1 Rationale

In the world at present, the shrinking land base and growing human population have heightened the challenges for forestry and forest products industries to source the needed types and quantity of trees. Therefore, sustainable development is being introduced to prevent the extinction of natural resources. Sustainable development must be based on environmental and economical aspects. Key areas for research and development are use of the changing wood resource, extension of the resource, and environmentally-friendly technologies. Technologies like wood-composite products and engineered wood products can address the increasing diversity of the wood resource. In addition, the wood resource can be extended through recycling – these technologies include using wood waste to produce new wood products such as making paper from wastepaper. Using these technologies, we can minimise environmental problems and increase the economic viability of sustainable forest management (Hamilton, 1997).

Throughout the world, particleboard holds 57 % of the total volume of solid panel product consumption (Drake 1995; 1997). According to an Australian forest statistics report, during the 17-year period from 1984-85 to 2000-01, the average annual rise in consumption of particleboard in Australia was 1.6 % (ANU 2002).

In addition, considerable quantities of hardwood sawmill residues are produced throughout Australia. In Victoria alone, over a million cubic meters of saw logs are converted into sawn timber per year, which produces in excess of 200,000 tonnes of hardwood sawdust which is

currently disposed of by burning. However, this leads to a number of environmental issues. (Nirdosha et al, 2007).

Numerous innovations and improved technologies have resulted in the development of many types of wood panel. For example, Guler and Kalaycioglu (2006) investigated the possibility of utilizing particles from Calabria pine (P. brutia Ten) and sunflower stalks in the manufacture of three-layer particleboards. Different proportions of sunflower stalks and Calabria pine wood were used to produce the particleboards, and various samples were produced and evaluated. It was found that a 50:50 mixture of Calabria pine wood and sunflower stalks produced the optimum configuration in terms of mechanical properties of the particleboard, which complied with the minimum standards for general grade particleboard. However, they observed that the board properties could be improved by the utilization of hydrophobic additives such as paraffin in the matrix. Finding this type of new application for sunflower stalks has the potential to reduce pressure on forest resources.

A study of an alternative material in particleboard industry was conducted by Gokay and Kalaycioglu (1997). The test particleboards were produced from the residues from a tea factory. The results were compared with British Standards BS EN 312:2003. Factors affecting the mechanical properties were found through experiments, and it was observed that increased amounts of formaldehyde in the adhesive caused higher formaldehyde emissions. Therefore, formaldehyde emission was a main concern of this study.

During 2005, a study conducted (Nirdosha et al 2007) succeeded in producing a particleboard from hardwood sawmill residue in the laboratory at RMIT University. This particleboard satisfies the AS/NZS requirements for physical and mechanical properties. However, this product utilizes about 10 % more resin than that of industry-made softwood particleboard and the density of this particleboard is higher than the density of conventional softwood boards. Therefore, further investigations are needed to optimize the properties of hardwood residue boards.

Many attempts have been made by researchers to improve and produce new particleboard products using different raw materials. All the researchers have had the same aim, which is the endeavour to prevent the extinction of forests. Therefore, it is worthwhile to continue further investigations on hardwood particleboard production.

The following sections of this chapter will discuss the aims and objectives of this investigation, followed by the outline of the thesis.

1.2 Aims

The major aim of this investigation was to develop new knowledge and a technology for improving and optimizing particleboard produced using hardwood sawmill residues. This required research into economic and environmental factors, studies of factors affecting properties of particleboard, the possibility and suitability of mixing hardwood and softwood flakes for board optimization, and identifying the optimum configuration for the mechanical and physical properties of boards.

1.3 Objectives

The objectives of the research were as follows:

- To confirm the feasibility of continuing previous work (Nirdosha et al, 2007). Analysis was carried out of the utilisation of hardwood sawmill residues, production costs and the environmental benefits of hardwood particleboard.
- To develop an understanding of factors (process variables and material variables) that affects the mechanical properties of particleboard.
- To develop an understanding of the effects of resin on particleboard.
- To produce a particleboard made of mixtures of hardwood and softwood sawmill residues, with mechanical properties complying with AS/NZS 1859.1:1997.
- To develop and validate composite material models to predict the Modulus of Elasticity (MOE), Modulus of Rupture (MOR), Internal Bonding (IB) and density of particleboard for a given set of process variables and material compositions, and to use these models to optimize board properties within a given process-parameter range.

1.4 Potential benefits

A study of this nature is important both to expand fundamental knowledge and enhance industrial applications. An outcome of this project will be a more economical particleboard which uses large quantities of hardwood sawmill residue, and which is expected to be readily accepted by the industry. It will also make a significant contribution to the sustainability of the Australian timber industry and the environment by reducing logging to produce flakes for softwood particleboard. The uses of hardwood residues will also be more beneficial and diverse.

1.5 Outline of the thesis

In order to achieve the objectives, a well planned research program was essential. The thesis is divided into twelve chapters, and a brief description of each chapter is outlined below.

1.5.1 Chapter 1: Introduction

Chapter 1 introduces the topic, rationale, aims and objectives of the research. It is also outlines the organisation of the thesis.

1.5.2 Chapter 2: Significance of the research project

In order to ensure the feasibility of continuing the previous work (Gamage et al. 2007), some preliminary analysis is provided in Chapter 2 covering the following aspects:

- Amount of hardwood sawmill residues being disposed of or utilized
- Market analysis of particleboard
- Cost analysis of hardwood particleboard
- Environmental benefits of utilising hardwood sawmill residue in particleboard

1.5.3 Chapter 3: Literature review

Chapter 3 reviews the literature on important physical and mechanical properties of particleboard and the effects of material and process variables on these properties. Current practices and the findings of previous research are also discussed.

1.5.4 Chapter 4: Design of experiments (DOE)

Large numbers of process variables affecting board properties are identified in Chapter 3. Due to these process variables, the theory of experimental design was considered as a possible method of planning and analyzing experiments. The theory of experimental design is thoroughly discussed in this chapter. The advantage of using experimental design based on factorial design is highlighted. The chapter also explains the analytical techniques used for data analysis (ANOVA).

1.5.5 Chapter 5: General procedure for producing particleboard in the laboratory and methods of testing

Chapter 5 illustrates the apparatus used in the laboratory to produce particleboards and the testing methods used to test board properties. The manufacturing procedure of particleboard in the laboratory is clearly explained, and the calculations of various mix proportions derived from experimental designs with a number of variables are presented.

1.5.6 Chapter 6: Particleboard production using mixtures of softwood and hardwood residues

After identifying the process and material variables for the particleboard production, a series of particleboards was made using mixtures of softwood and hardwood residues in different proportions. In this chapter, a mixture of hardwood and softwood is applied in both core and surface layers at the portion of 40 % or 60 % of total particles in each layer. Boards produced were tested and analyzed, and the chapter identifies the most influential parameters on the

properties. These results are compared with the results of previous research (Nirdosha et al. 2007).

1.5.7 Chapter 7: Investigation of particleboard production using mixtures of softwood and hardwood in core and hardwood in the surface

From Chapter 6, it was observed that hardwood particles in the surface contribute to bending strength, and some adjustment of moisture content is essential for surfaces and core layers in order to achieve apparent non-uniform moisture distribution. In this chapter, two hypotheses are established in accordance with the findings of Chapter 6. A new series of particleboards are produced using hardwood only in the surface and a mixture of softwood and hardwood in the core. The analysis of results is completed with the assistance of MINITAB. Normal probability plot, Pareto chart and contour plot are applied to recognize the significant factors and relationships between variables with board properties. Further discussion of the hypotheses is also provided.

1.5.8 Chapter 8: Identifying appropriate configuration of hard/softwood particles in core and surfaces

Based on the analysis of results in Chapter 7, it was confirmed that the hypothesis of hardwood particles only in surface contributes to bending strength was not justified. In this chapter, in order to obtain an optimal mixture of hard/softwood in particleboard production, a series of particleboards with different hard/softwood mixture configurations in core and surfaces are tested with a different experimental design to the preliminary study. Constant process variables are applied on each individual board, and this chapter presents the results of this third series of experiments.

1.5.9 Chapter 9: Particleboard production using the optimized S – H configuration

From the Series 3 experiments presented in Chapter 8, particleboard produced with S – H configuration (softwood only in surface and hardwood only in core) was found to be the most suitable mixture configuration of hard/softwood for particleboard production. In this chapter, four important process variables are selected for investigation. Eight particleboards were made in the laboratory and then analyzed in accordance with the requirements of AS/NZS. The results are used to develop polynomial regression models to predict the board properties (MOR, MOE, IB and Density) as functions of processing parameters. This chapter presents the details of the experimental and analytical study. The validation of these models is also discussed, and the validated models are then used for board optimization.

1.5.10 Chapter 10: Surface studies and thermal analysis of hardwood and softwood composites

The internal conditions of particleboard, such as the penetration level of resin into wood particles, the resin curing point, the resin curing level and the composition of particles, require further investigation in order to obtain a better understanding of the internal structure of and reactions within particleboard. This chapter reports some studies carried out to gain some fundamental knowledge of the internal structure of particleboard. GADDS mapping is used for the surface analysis of hardwood and softwood particleboard, and TGA is applied to understand the composition of hardwood and softwood particles using thermal analysis. Chapter 10 presents this analytical work, and outlines possible future studies.

1.5.11 Chapter 11: Conclusions and recommendations

Chapter 11 summarizes the general conclusions of the work reported in the thesis, and provides recommendations and suggestions for future work.

Chapter 2

SIGNIFICANCE OF THE RESEARCH PROJECT

2.1 Overview

This chapter presents a preliminary study of the current industry needs to ascertain the significance of the research area. It covers the demand for and consumption of particleboard, the need for new raw materials, industry expectations, and the environmental and economic benefits of using hardwood residues as raw material for particleboard.

2.2 Introduction

One of the greatest challenges of the forestry sector and the global wood industries is the need to address and overcome declining raw material supplies, to fully utilize available resources, and to take advantage of innovations in wood processing. A growing number of timber producers are facing raw material shortages and some wood processors are now operating below 50 % of capacity. This problem is expected to worsen in future.

In this situation, logging and mill residues are emerging as a major potential source of raw material. It is hoped that more efficient use of residues can lead to a reduction in the areas logged every year. In addition, the disposal of waste and residues is now prohibited or very costly, and after a catastrophic fire caused by the burning of waste and residues, restrictions on open burning have been enforced more strictly in some countries (Dykstra, 2001). Although some wood processors and power plants chip and burn residues to produce steam

and electricity, strict environmental regulations concerning waste disposal and fire risks restrict the burning of residues.

In 1998, the Asia-Pacific Forestry Commission (APFC) urged the Food and Agriculture Organization (FAO) to coordinate a review of residue availability and the potential implications. Other international organizations are also supporting complementary work in this area.

A recent research project undertaken at RMIT University explores the feasibility of utilizing large quantities of hardwood sawmill waste in making particleboard. This chapter presents the outcomes of the early research and an economic analysis of particleboard production undertaken to establish the future directions for the research.

2.3 Demand for and Consumption of Particleboard

2.3.1 Global Particleboard

Particleboard is in the midst of a phase of expansion in some regions, but growth remains modest in others. There has been rapid growth in China, Russia, Turkey, other East European countries, Southeast Asia and South America over the past five years. Growth in North America, Europe, Japan and Australasia has been modest over the past five years, although capacity utilization was relatively high until the onset of the financial crisis and economic downturn.

BIS Shrapnel (2010) reported that the economic downturn in the United States reached the bottom of the cycle in 2009, and is expected to move into a rapid upswing to 2013. Global production of particleboard is forecast to increase from 69.9 million cubic metres in 2009 to 84.1 million cubic metres in 2013.

In 2009, Europe produced sixty two per cent of the world's particleboard, North Asia produced fourteen per cent, North America produced nine per cent, South Asia produced eight per cent, South America produced six per cent, and Australasia produced one per cent. Production of particleboard in Europe is forecast to drop slightly to sixty per cent by 2013.

Over the same period, production in North America and South Asia is expected to increase slightly to nine per cent, and in South America production is forecast to increase to seven per cent. Production of particleboard in North Asia and in Australasia will remain unchanged.

Announcements have been made by several plants in key particleboard production regions confirming the expanded capacity for their particleboard plants. Particleboard is also a potential substitute for Medium Density Fibreboard (MDF) and plywood as it is a more cost-effective alternative for furniture manufacturing. (BIS Shrapnel, 2010)

According to Drake (1997), in 1994, global consumption of particleboard was 57 % of the volume of total panel product consumption. Worldwide demand for particleboard has been growing steadily at a rate between 2 % and 5 % per annum, and according to current Australia forest statistics, particleboard consumption in Australia increased by 7 % during year 2001. However, there was a decline of global particleboard consumption by five percent in 2009 due to the global financial crisis. Particleboard consumption is expected to grow at the rate of 6 % per annum from 2010 to 2013 with the economic recovery from the global financial crisis. These figures indicate that the market demand for particleboard has the potential to grow (FAO, 1997).

2.3.2 Particleboard Consumption in Asia-Pacific Region

According to FAO (1997), particleboard consumption in the Asia-Pacific region grew at a rate of 15 % per annum from 1990 to 1995. It expanded from 4.9 million m^3 in 1990 to 9.9 million m^3 in 1995. The production of particleboard in the region grew from a level of 4.2 million m^3 in 1990 to 9.6 million m^3 in 1995, a very healthy growth rate of 18 % per year. However, the production is inadequate to keep up with the growth of demand.

China is the largest particleboard producer and consumer in the Asia-Pacific region (FAO, 1997). China's production of particleboard increased from 1.5 million m^3 in 1990 to 4.4 million m^3 in 1995.

Japan has also expanded its particleboard production to meet their particleboard consumption. In 1990, their production was at 1.0 million m^3 with consumption at 1.17 million m^3; while production increased to 1.25 million m^3 with the consumption of 1.65 million m^3 in year 1995. It is clear that Japanese supplies still do not meet their needs.

In 1997, FAO forecast particleboard demand to increase to 29.6 million m^3 by the year 2010. In order to meet this forecast demand, an average increase of 1.3 million m^3 production capacity per year was required.

2.3.3 Particleboard Production and Consumption in Australia

The production and consumption of particleboard have been growing in Australia. From 1991 to 2006, the production and consumption of particleboard both increased at an average rate of around 2.3 % per annum and 2.8 % per annum respectively, as shown as Figure 2.1.

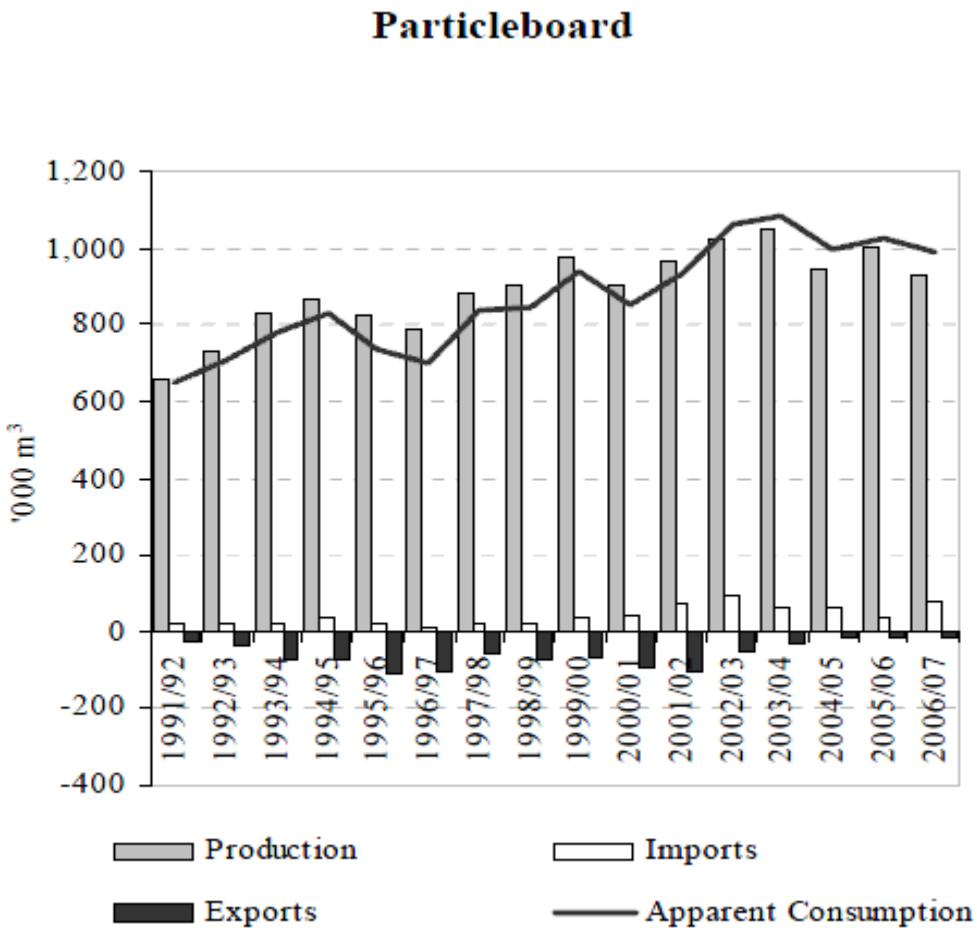


Figure 2.1 Production and consumption of particleboard in Australia (ABARE, 2006)

The increasing consumption of particleboard has been driven by its substitution for solid wood in a range of markets, such as furniture and construction.

Most particleboard mills in Australia are relatively old and are generally considered to have high production costs. While domestic production capacity has matched consumption closely,

ongoing growth in the market could provide opportunities for more competitive facilities. Increasing competition for residual wood from MDF, wood chip exports and other potential uses are likely to constrain investment in new particleboard production. One potential option might be a new particleboard production facility based on wood not currently utilised, such as residues from logging operations or recycled wood.

2.3.4 Availability of sawmill residues

According to Enters (2001), the amount of residue produced per year in sawmilling in a number of selected countries is about 42 million m^3. Among these countries, approximately 90 % of the sawmill residues is generated in only four main countries, China, India, Indonesia and Malaysia. This percentage was even greater before the economic crisis of 1997. Total residue volumes prior to the crisis were probably 30 % to 40 % higher.

The total volume of sawmill residues is not the volume that is currently available since much larger percentages of sawmill residues are already being utilized. Especially in some areas where wood is in short supply, residues are unlikely to be 'waste', but are likely to be claimed for further industrial processing. For example, in the State of Sarawak, Malaysia, sawmill residues are used to the extent that shortages of raw materials are imminent locally.

In Australia there is still a significant amount of scrap materials generated by sawmill and other wood processing factories. 200,000 m^3 from saw logs and 300,000 m^3 from pulp logs are considered hardwood residue or wastes. According to Gamage et al (2005) annual hardwood residue production is approximately 200,000 tonnes. The residues are sold for kiln heating or gardening purposes. A tonne (2.5 m^3) of hardwood residue from Dormit Pty Ltd. is sold for only 1.50 dollars, although the price may range from $1.50 to $30 depending on the location of the sawmill.

2.3.5 Potential utilization of sawmill residues

Many sawmills are retooling in order to raise the performance and productivity of breakdown saws and re-saws. This can increase the recovery rate and minimize waste. However, even substantial attempts would likely reduce sawmill residues by only 5 % to 10 %. Thus, the best solution is to fully utilize the residues.

Wan et al. (1999) suggest that any wood-waste management strategy should follow the '4Rs' as shown below:

1. Reduce
2. Reuse
3. Recycle
4. Recover

These writers categorize potential utilization into three broad categories:

1. Energy production
2. Secondary raw materials to be used by wood-based industries
3. Secondary raw materials to be used by industries outside the wood industry

For the wood composites industry, sawmill residues of mixed hardwood species can form an important raw material, especially for the chip and board industry. This is attractive for large-scale uses where mills have opportunities to sell their unwanted waste.

From 1990 to 1997, world production of five major wood-based panels increased from 106 million m^3 to 150 million m^3. However, due to the downturn of many Asian economies, consumption and production fell in 1998. Plywood was most affected by the economic crisis. However, particleboard and MDF producers registered slight increases. This difference can probably be explained by the different raw material requirements. Particleboard and MDF can actually make use of much cheaper raw materials, such as sawmill residues. In addition, there may be a price difference between plywood and particleboard. Therefore, demand for particleboard will be slightly higher in comparison to plywood.

According to the FAO (1997), from the 68,510 million m^3 of wood residues, the Asia-Pacific is capable of producing another 50 million m^3 per year of particleboard, MDF or other fibreboards.

Therefore, the future role of wood residues as raw material for the expanding particleboard and MDF industries is likely to increase.

2.4 Current Research into Making Particleboard Using Sawmill Waste

Hardwood sawmill residues have not been investigated for particleboard production due to their inherent properties such as their high density, higher extractive content and higher moisture content. However, the move in recent years by the sawn-wood industry towards the harvesting and processing of re-growth and plantation resources has opened up new opportunities for both the residue generators and potential users such as the particleboard industry, since the residues are likely to have a lower extractive content and density. Therefore, a research initiative at RMIT University was carried out to ascertain whether hardwood residues can be used in producing three-layer particleboard which satisfies the AS/NZS standards for physical and mechanical properties (Nirdosha et al, 2007).

A multivariate factorial experimental design was developed using seven material and process variables with two levels to investigate the effects on the mechanical and physical properties of the finished boards. The target density of the board was 710 kg/m^3 and the target thickness was 15.2 mm.

Table 2.1 below shows the experimental variables and results from hardwood particleboard; Table 2.2 indicates the estimated effects and coefficients for the physical properties of a board.

Board number	Moisture (%[a])	Resin surface (%[b])	Resin core (%[b])	Hardener (%[c])	Pressing time (s)	Press temperature (e)	IB (kPa)	MOE (MPa)	MOR (MPa)	Thickness (mm)	Mean density (kg/m^3)	Surface density (kg/m^3)	Core density (kg/m^3)
ST 1	11	8	5	1	120	150	74.40	577	5.801	16.92	679.870	605	494
ST 2	11	20	5	3	300	200	389.32	1681	12.12	15.20	713.374	904	676
ST 3	22	8	13	3	300	150	330.12	1386	10.32	15.64	750.733	911	679
ST 4	11	8	13	3	120	200	320.00	1107	11.22	16.40	708.058	873	655
ST 5	22	20	13	1	120	200	545.88	2088	16.59	15.52	723.797	985	665
ST 6	11	20	13	1	300	150	605.32	1743	13.7	15.62	740.845	866	690
ST 7	22	8	5	3	120	200	86.12	810	6.861	16.52	690.377	737	578
ST 8	22	20	5	1	300	150	282.92	1650	11.62	15.40	716.508	887	624
ST 9	11	20	13	3	120	150	368.52	1240	9.58	16.74	668.346	759	583
ST 10	22	8	5	1	300	200	76.28	999	7.29	15.74	711.330	812	555
ST 11	22	20	13	3	300	200	384.00	1356	10.00	15.20	717.140	915	677
ST 12	22	8	13	1	120	150	122.40	565	6.02	19.52	603.000	611	512
ST 13	11	8	13	1	300	200	638.92	1357	11.07	15.10	713.267	756	611
ST 14	11	20	5	1	120	200	415.72	1069	8.01	16.28	698.083	760	553
ST 15	11	8	5	3	300	150	103.20	820	6.93	17.46	637.610	792	668
ST 16	22	20	5	3	120	150	17.60	371	3.73	19.40	608.162	473	462
AS/NZS 1859 (2004) requirement for general purpose particleboard							400	NA	16.00	NA = Not required			
AS/NZS 1859 (2004) requirement for moisture resistance general purpose particleboard							400	2400	16.00				

[a] % based on moisture weight per weight of oven dry wood residue.
[b] % based on weight of resin solid per weight of oven dry wood residue.
[c] % based on weight of hardener solid per weight of resin solid used in that layer.

Table 2.1: Experimental Variables and Results of Hardwood Particleboard (Nirdosha et al, 2008)

Term	MOE		MOR		IB		Thickness		Surface density		Mean density	
	T	P	T	P	T	P	T	P	T	P	T	P
Moisture surface	−0.94	0.393	−6.37	0.003	−6.82	0.001	2.81	0.038	0.07	0.948	−0.67	0.537
Moisture core	−1.23	0.274	−0.94	0.4	−4.28	0.008	7.34	0.001	−1.96	0.145	−4.07	0.015
Resin surface	7.74	0.001	17.57	0	8.01	0	−3.42	0.019	2	0.139	1.62	0.181
Resin core	0.23	0.831	4.45	0.011	11.91	0	−2.6	0.048	3.13	0.052	2.98	0.041
Hardener core	−4.88	0.005	−10.18	0.001	−4.86	0.005	2	0.102	0.36	0.74	−1.63	0.178
Pressing time	2.26	0.073	4.03	0.016	5.48	0.003	−10.18	0	4.61	0.019	5.64	0.005
Press temperature	5.53	0.003	11.17	0	6.06	0.002	−9.17	0	3.71	0.034	4.75	0.009
Moisture surface x moisture core	−2.65	0.045	−3.61	0.022	−3.94	0.011	4.13	0.009	−4.32	0.023	−4.42	0.012
Moisture surface x press temperature	3.37	0.02	7.45	0.002	−1.74	0.142	−2.64	0.046	1.31	0.281	NS	NS
Moisture surface x hardener core	NS	NS	−2.96	0.041	NS	NS	NS	NS	−2.66	0.076	NS	NS
Moisture surface x resin core	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS
Moisture core x resin core	NS	NS	NS	NS	NS	NS	NS	NS	0.66	0.554	NS	NS
Moisture surface x pressing time	NS	NS	NS	NS	NS	NS	−5.01	0.004	1.76	0.176	3.86	0.018
Moisture core x resin surface	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	2.05	0.11
Moisture surface x moisture core x resin core	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	−3.37	0.028

NS = not significant.

Table 2.2: Estimated effects and coefficients for physical properties of a board (Nirdosha et al, 2008)

MOE, MOR, IB, mean surface density, mean density and thickness are shown in Table 2.1. Table 2.2 shows the statistical analysis, incorporating the theory of experimental design to calculate the test statistical values (T) for each process variable and the probability of null hypothesis (P). The effect that is not significant is normally distributed with mean zero and variance ∂^2. A variable with a significant effect will have a higher "T" value compared to non-significant variables (Myers and Montgomery, 2002). Therefore, parameters with higher

"T" values are considered as significant variables. The level of significance of the variable considered is estimated by the probability of the null hypothesis (P). If $P \leq 0.05$ for any process variable with respect to any board property, that process variable has a significant effect on that board property with 95 % significance level.

It was found that moisture is a critical variable that needs to be controlled carefully to achieve better compaction, and to transfer heat and mass to the core of the board during hot pressing. Moisture has a negative effect on the rigidity of the board (MOE) as well as the IB.

However, the interaction of surface moisture with press temperature increased board flexural strength (MOE and MOR). Besides, resin content and pressing time are significantly affecting both mechanical and physical properties since it was obtained that hardwood particleboard have better properties when the resin content and pressing time are higher.

However, some problems were observed in the previous investigations (Nirdosha et al, 2008). For example, it was noted that the density of hardwood particleboard was considerably higher than the density of conventional softwood particleboard, and higher amounts of resin are needed for the surface layer to achieve the same strength properties as softwood particleboard. Mixing hardwood residues with softwood flakes at the surface layer was proposed in order to reduce the particleboard density and resin content.

It was also found that the pressing time required is about 20 % more than the industry best practice for softwood particleboard production. Since resin consumption and pressing time directly control the production cost of particleboard, investigation into the efficiency of resin is essential. Alternative resins such as melamine-urea formaldehyde or MDI require to be studied. These superior resins may reduce the amount of resin required as well as the curing time.

2.5 Production cost of particleboard

The particleboard production process adopted at RMIT University is similar to the process adopted in using custom-flaked softwood. Therefore, an evaluation of the net profit from hardwood particleboard can be made by reference to a softwood particleboard plant. The major differences between the new board product and softwood boards is that the new

product has a higher resin consumption, a higher density and a longer pressing time to achieve mechanical properties equivalent to softwood boards.

Based on advice from Justin Tonks (Production Manager of D&R Henderson Pty. Ltd), the pressing time of 120 seconds in making a hardwood particleboard would not be a major factor on the particleboard production cost since the standard press times for a 16 mm standard grade panel would generally be around 110 seconds. Density would not be an issue if the hardwood particleboard is marketed as a new option in the wood-based panel industry. Therefore, the major concern of the hardwood particleboard production would be the resin content. It has been found that a higher amount of resin is needed for the surface layer to achieve the same strength properties as softwood particleboard. Hence, higher resin consumption needs to be considered for the evaluation of a hardwood particleboard plant.

	Plant Capacity M^3	Capital cost $million	Yield %	Cost of raw materials		Cost of wood raw materials $ million	Other costs $million	Net Profit $million
				Resin $million	Wood chips $/tonne			
Softwood particleboard	100,000	25	75	7	100	9.07 +7 = 16.07	14.07	2
Hardwood residue boards	100,000	25	75	8.75	30	2.84 +8.75 =11.59	14.07	6.48
Hardwood residue boards – with adjusted price of raw materials	100,000	25	75	8.75	30 + 30 (transport cost assuming max. of 300 km)	5.68+ 8.75= 14.43	14.07	3.64
Worst case – price of hardwood residue increase to $100/tonne	100,000	25	75	8.75	100	9.47+ 8.75= 18.22	14.07	1.6

Table 2.3: Analysis of production cost

The density of softwood wood chips is generally around 680 kg /m^3. Therefore, a 100,000 m^3 capacity plant will need 100 x100, 000 x 680/1000/0.75 tonnes assuming a yield of 75 %. A similar calculation based on the higher density of 710 kg/m^3 of hardwood residue will

produce30 x 100,000 x 710/1000/0.75. The worst case would be an increase in the hardwood residue price to $100 per tonne, which would create a reduction in profit. Whilst the analysis given in Table 2.3 is simplified, it enables an appreciation of the issues associated with hardwood boards.

The most realistic scenario would be the hardwood boards with an adjusted price of raw materials (Row 3 of Table 2.3).

As discussed earlier, annual hardwood residue production in Victoria is approximately 200,000 tonnes. Upon introduction of a hardwood particleboard plant, hardwood sawmillers will be able to make additional annual incomes of $30 x 200000 = 6 million dollars. Furthermore, particleboard manufacturers will be able to widen their market by developing new products. As a result, Australia's demand for particleboard could be met by local suppliers instead of importing.

2.6 Environmental Benefits

In considering the environmental benefits of the new application, the following scenarios may be considered:

Scenario (a): Decomposing wood residue as a fertilizer or a similar application. This will release the same amount of CO_2 into the atmosphere as the trees have absorbed from the environment. In addition, a significant amount of CO_2 has also been released to the atmosphere at the saw mills.

Scenario (b): Disposing of wood residue by burning. Burning of residue will increase the emissions significantly over those already emitted at the sawmill

Scenario (c): Using residue in particleboard. The residue is captured in a carbon sink (a board product which lasts longer than 50 years). Therefore there is a negative emission of carbon dioxide which helps to balance the carbon equation in relation to the sawmill industry.

Scenario (d): Burning residue and generating energy

In order to assess the environmental effects, an attempt is made to calculate the total CO_2 emission from the burning of residue.

The estimated CO_2 emissions from a sawmill plant of 20,000 m^3 capacity are reported to be 3550 tonnes per year. Noting that 25 % of the capacity is wood residue, the above operation would have emitted 887.5 tonnes of CO_2 in making the residue (Goble et al. 2007). Burning the residue will further release CO_2 into the environment. The carbon dioxide emissions can be calculated as:

$qCO_2 = cf / hf\ CCO_2/C_m$ (The Engineering ToolBox, 2005)

Where:

qCO_2 = *specific* CO_2 *emission (CO2/kWh)*
cf = specific carbon content in the fuel (kgC/kgfuel)
hf = specific energy content (kWh/kgfuel)
Cm = specific mass Carbon (kg/mol Carbon)
CCO_2 = *specific mass Carbon Dioxide (kg/mol* CO_2*)*

The above calculation gives the combustion outcome of wood residues as approximately 0.18 kg/kWh of CO_2 (The Engineering ToolBox, 2005). The energy equivalency of wood residue in Australia is estimated to be 120 million MJ (Goble et al. 2007). Assuming that 50 % of the residue is burned, the release of carbon dioxide would be 10.8×10^9 tonnes of CO_2 per year.

Forestry residues are a source of biomass. When biomass is burned, it releases energy, generally in the form of heat. The energy obtained from biomass is a form of renewable energy. Using biomass energy maintains the total amount of carbon dioxide in the environment, unlike burning fossil fuels which increases the amount of carbon dioxide in the air. Of all the existing renewable energy sources, biomass is the only one that stores solar energy as a convenient solid, liquid or gaseous fuel (Twidell, 1998). However, there are some environmental constraints on biomass use. If the wood is not fully burnt, small particles of soot-like material (which can cause smog on a large scale), carbon monoxide and other organic gases are produced. If wood is burnt at high temperatures, oxides of nitrogen are produced which can cause photochemical smog. Therefore, in developing countries, people have health problems from the air pollution. Deforestation and serious ecological and social

problems will occur if natural forests are overused as a source of wood for fuel (Twidell, 1998).

The production and processing of biomass may require a significant amount of energy input. Therefore, the amount of energy produced minus the amount of energy used in its production must be calculated in order to optimize the biomass-to-energy conversion process. At a 30 % conversion rate, burning of wood residue will have the associated benefit of making 36 million MJ of green energy.

In contrast to the utilization of sawmill residue as a biomass, particleboard production from sawmill residues is more beneficial since it would not lead to the environmental constraints discussed above. According to the CSIRO, Australia, wood (especially from sustainable harvesting practices) has been shown to be the best material for many different applications, as it has a minimal impact on the environment compared to other materials such as metals and plastics. Wood has been found to be superior to either metals or plastics. The use of even small amounts of other materials (such as metal or plastic trim, glues and varnishes) drastically increases the environmental impact (Taylor et al, 2003).

Furthermore, increasing the use of wood material in construction is also a potential option for reducing net carbon dioxide emissions. This is because of the relatively low energy needed to manufacture wood products compared with alternative materials. In comparison with concrete-frame building, wood-framed construction requires less energy, and emits less CO_2 to the atmosphere (Gustavsson et al. 2005).

Scenario (c) above has many environmental benefits, which would be adequate to cover the additional cost of raw materials.

2.7 Summary

Conclusions drawn from the work presented in this chapter can be summarized as follows:

- Production of a particleboard which satisfies the current standards for mechanical properties using 100 % sawmill waste is feasible
- There is significant growth expected in the global consumption of panel products. Therefore, the new board product proposed will meet the current demand.
- Economic analysis of the particleboard production process clearly indicates that the higher resin consumption is offset by the low cost of raw materials, except in the situation where the cost of hardwood residue escalates to be similar to that of softwood flakes.
- The new product also offers significant environmental benefits which can overcome the additional costs incurred in requiring 25 % more resin.
- A study of the feasibility of reducing the production cost using mixtures of softwood and hardwood is timely and relevant.

Chapter 3

LITERATURE REVIEW

3.1 Overview

Prior to the design of a research program covering particleboard made of mixtures of hardwood and softwood, a review of literature was undertaken to establish the current practice, the findings of previous research, the major variables affecting particleboard production and appropriate experimental methods.

3.2 Introduction

Particleboard is an engineered wood product manufactured from wood species, such as wood chips, sawmill residues, or saw dust, which is bound with appropriate resin and then pressed and extruded.

In the literature review, the basic process of making particleboard needs to be focused on since there is a lack of detailed experimental procedures in most of the published reports. In some cases, the experimental details consist only of a statement that particleboard was made in the laboratory, without any information of particle type, adhesive content and pressing details.

Various particleboard physical properties are not readily classified as either strength or hygroscopic characteristics, but are important for most particleboard applications. Many of these properties, such as density, vertical density gradient, and spring-back, have a significant

influence on the strength and hygroscopic characteristics of the particleboard panel. However, these, as well as formaldehyde release and surface characteristics, are also dependent upon processing parameters such as pressing conditions, resin content, and particle geometry. It will be helpful to consider these physical properties and various processing parameters in this section.

3.3 Particleboard Production Process

This section outlines the general methodology of the particleboard production process. The first step of the process is particle preparation. Custom-flaked wood particles are the major raw material of particleboards used in Australia. They are normally prepared either on-site or off-site. The particles are produced by a diverse range of reduction equipment which is matched to the variety and size desired. The commercial particleboard industry uses two different types of wood chips for the core and surface layer of particleboard.

The particles then need to be dried so that the overall moisture level of the particles is in the order of three to eight percent for the purpose of bonding with liquid resins. Directly after drying, the particles are screened for size in vibrating screens. Particles are separated according to size, for the purpose of grading the particle for the surface and core layers.

Adhesives in the form of urea, phenol and melamine formaldehyde are generally used to bind the particle mix. Between three and ten percent by weight of resin, together with additives used to improve resin properties, are blended under batching conditions. Surface and core layers are blended separately from two different recipes.

The glue-coated particles are formed into a mat before the pressing process takes place. Pre-pressing or cold pressing is used to reduce the thickness of the mat. This is an important step where pre-curing of particles could occur if the surfaces of the mat are cured before being fully consolidated when the press is loaded.

The formed mats of glued particles and fibres are transferred to the hot press for pressing and curing. This operation is critical and requires carefully controlled heat, pressure and timing. Typical pressures are 2-3 MPa, temperatures 140-220^{o} C and press times 6-15 seconds per mm of board thickness. The thick mats are compressed in the press with the thickness being

controlled either by thickness bars (stops) or press travel distance. As soon as heat is applied, the glue curing process begins and full pressure is quickly applied to reach the desired thickness before cure. Full pressure at stops is held for the prescribed time, then pressure is slowly reduced until the pressure is released. Airing cycles are important to allow steam generated to escape, thus preventing 'blown boards'.

The boards are finally taken from the press and left to cool. In the final stage, the boards are trimmed to the required size and then sanded to the required thickness.

Figure 3.1 below shows the layout of a continuous industrial particleboard production plant.

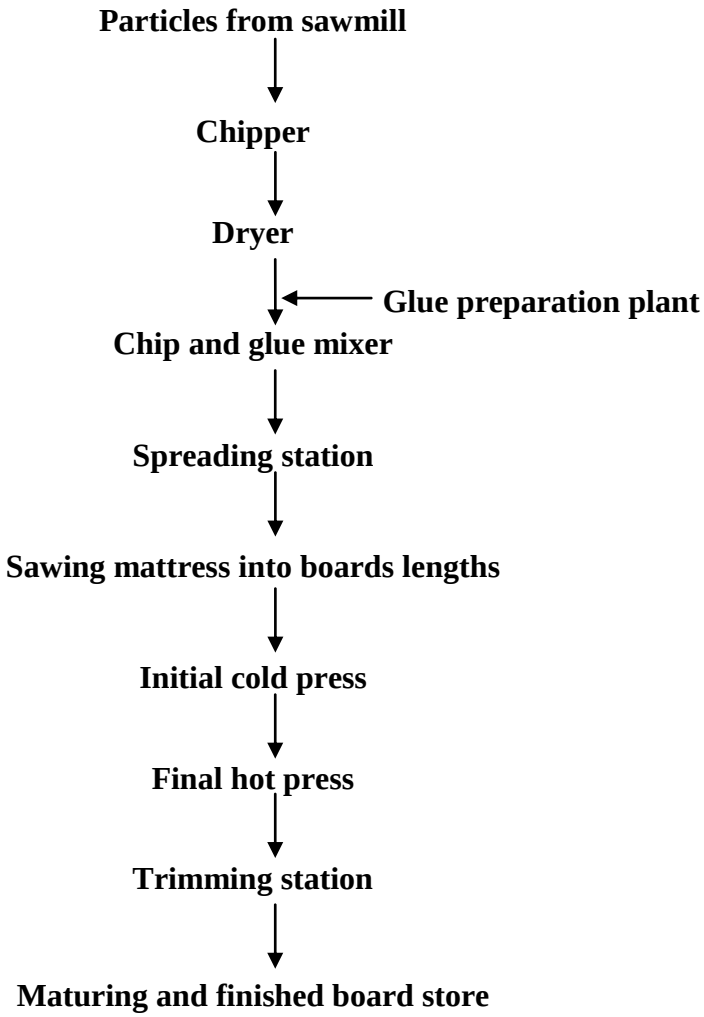


Figure 3.1 Layout of particleboard production process

3.4 Physical and Mechanical Properties of a Particleboard

3.4.1 Density

Density is a measure of the compactness of the individual particles in a board. The average final density of a particleboard is dependent upon the raw material density and the compaction of the mat in the hot press. Any change in one of these factors requires an adjustment of the other if the board density is to remain constant. Therefore, the average board density can be changed by adjusting one of these factors. However, higher density panels produced by increasing the compaction level will not have properties equal to the same density board produced with a higher density wood furnish.

The pressing operation consolidates the particle mat to the desired density or thickness and polymerizes the binder system between individual particles. Therefore, the amount and condition of the material in the mat, together with the pressing techniques, determine the final board density. Thickness control of particleboard is attained by using 'stops'. The maximum pressure employed for particleboard in a press equipped with 'stops' controls the rate of press closing, which compacts the mat to the desired thickness. The rate of press closing does not affect the final board density as long as the press temperature and pressing time are sufficient to cure the resin and allow evaporation of excess water. The average board density is dependent upon the quantity and density of the wood furnish used to make a particleboard of a given thickness.

Apart from the amount of wood furnish in the mat, the final density of particleboard is also dependent upon processing conditions prior to the pressing operation, such as furnish species, preparation and drying, resin content and other additives.

The effect of furnish species on particleboard physical properties is primarily related to the wood density and the necessity of compressing the wood to obtain satisfactory board density. According to Myron (1977), particleboard with a lower density than the density of the wood furnish will be unsatisfactory. Therefore, a compaction of the mat to a higher density than the furnish density is required. This will allow better surface contact between the component particles of the mat. More adhesive-coated particles will be in intimate contact with other wood particles instead of with voids. This results in better adhesive utilization. Larmore

(1959) found that boards pressed under a high-compaction ratio (board density: species density, 1.6:1.0) showed higher MOR and MOE (bending stiffness of the board divided by the geometric moment of inertia) than did panels made with a lower-compression ratio (1.2:1.0) at the same board density. This further illustrates the necessity of compressing the wood to some extent to obtain satisfactory board properties.

Suchsland (1959) stated that the distribution of total particle thickness varies randomly in a mat. The region with greater total particle thickness experiences higher compaction than the area with low total particles. This can maintain a uniform thickness or compaction during the compression. Therefore, compression area is a significant factor that influences the bending strength and density of particleboard.

3.4.2 Spring-back

Spring-back is the non-recoverable thickness swelling that occurs when a finished particleboard is exposed to elevated humidity, or liquid water, and is subsequently returned to the original humidity and temperature condition. The additional thickness swelling in particleboard exposed to moisture greater than that normally expected for wood material, is due to the release of residual compressive stresses imparted to the board during the pressing of the mat in the hot press. The moisture content reduction while the mat is restrained in the hot press reduces the plasticity of the wood and results in a semi-permanent “set” of these compressive stresses. However, at any future date when the moisture content increases, the additional moisture will plasticize the wood and allow these stresses to be relieved, allowing expansion in the thickness direction.

Spring-back is considered one of the greatest deterrents to the use of particleboard in exterior construction where high equilibrium moisture content conditions exist. Therefore, there have been many attempts to develop methods to effectively stabilize particleboard from dimensional changes resulting from moisture uptake. Several research reports pertaining to spring-back studies will be reviewed in this section.

The present commercial attempts at keeping spring-back to a minimum use a greater amount of adhesive (6 % to 10 % solid by weight) than is actually necessary for adequate bonding (4 % to 6 %). According to Childs (1956), in regard to the effect of chip width, resin content

and board density on the spring-back of urea-formaldehyde bonded particleboards, spring-back decreases with increasing resin content. As the resin content increases, the component particles are stabilized, and spring-back will be reduced. It was also observed that spring-back increases with increasing board density as more compressive force is imparted to the particles which will spring-back on rewetting.

Mikhailov and Ostapenko (1972) also attributed the spring-back of particleboard to a non-uniform moisture content distribution in the thickness direction. They recommended lengthening the press time and reducing the mat moisture content to alleviate this tendency. This practice should reduce the moisture gradient from surface to core in the final panel, but the moisture gradient would normally be expected to contribute in only a minor way to particleboard spring-back.

Other methods of increasing stabilization and reducing spring-back have been reported as shown below (Mikhailov and Ostapenko, 1972):

- Treating with high temperatures after manufacture
- Treating with high temperature saturated steam after manufacture
- Acetylation of dry particles prior to pressing
- Impregnation of particles with polyethylene glycol prior to pressing
- Addition of phenol formaldehyde impregnating resins to dry particles prior to pressing

However, these approaches to the reduction of spring-back have not been adopted commercially because of cost or limited effectiveness.

3.4.3 Strength Properties

Strength properties play an important role in determining the grade of a particleboard. The various processing parameters have various effects on the resultant panel strength properties. For instance, Halligan and Schniewind (1974) reported the moisture content of board has a significant effect on board strength properties. Modulus of Rupture (MOR), Modulus of Elasticity (MOE) and Internal Bonding (IB) are the most significant strength properties that need to be reviewed.

3.4.3.1 Modulus of Rupture (MOR)

The modulus of rupture (MOR) is an important property determining the applicability of particleboard for structural components. The MOR of particleboard is dependent on various parameters, such as board density, particle configuration and orientation, material types and processing conditions.

Board density is the most widely-reported parameter that affects the MOR of a particleboard. Vital et al. (1974) found that MOR and MOE increased linearly with increased wood density and particleboard density. Many researchers have found that there is a high correlation between compaction ratio (board density divided by the wood furnish density) and MOR of final board (Hse 1975; Howards 1974; Vital et al. 1974). Vital et al. (1974) reported that a board pressed under a higher compaction ratio obtained a higher MOR. However, boards made to the same compaction ratio from different species density did not result in constant MOR values (Hse 1975). MOR was found to decrease as the species density increased (Stewart and Lehmann 1973). Obviously, compaction ratio is directly proportional to the particleboard density for furnish with constant specific gravity. Most studies have reported an increase in MOR with increasing board density as the compaction ratio increases the MOR.

MOR values are highly dependent upon the vertical density profile. By changing the processing variables, a wide change of vertical density profiles will be obtained. Therefore, MOR will change. Rice (1960) increased the MOR by 18 % by increasing the mat moisture content in the press from 9 % to 15 % for flake board. This is because higher moisture content increases the compressibility of the mat surface during press closing, resulting in increased surface densification and higher MOR values (Strickler 1959).

It is also important to consider the effect of particle configuration and orientation on MOR. According to Myron (1977), particle thickness and particle length influence the MOR values. Myron (1977) reported that particle thickness has more influence on MOR values than particle length. At lengths greater than 1 to 2 inches, significantly lower MOR values were found as the particle thickness increases above 0.02 inch.

Large particles can be satisfactorily bonded at lower resin contents, because the particle surface area is drastically reduced as the particle size increases. Gertjejansen and Haygreen (1973) compared the effect on adhesive consumption by using 1.5 inch long wafers and 0.5 inch flakes respectively. They found that MOR was reduced by approximately 9 % when

wafers were used instead of flakes. However, the adhesive content of waferboard was only 3 percent, compared to 8 % for the flake board. Consequently, significant adhesive savings are possible with larger particles, providing other properties are maintained.

Suchsland (1959) stated that ideal particle configuration for a three-layer particleboard is small particles for smoothness in particleboard surfaces, and large particles with less adhesive in the core.

3.4.3.2 Modulus of Elasticity (MOE)

The modulus of elasticity (MOE) is an important property as it is a measure of stiffness, or resistance to bending, when a material is stressed. In general, modulus of elasticity and modulus of rupture are affected similarly by various processing parameters. Hse (1975) found that particleboards of constant average density posses higher MOE values as the wood density decreases and as the compaction ratio increases.

Report from Lehmann (1970) significantly suggested a direct relationship between board density and the effective MOE and concluded that an increase in density while maintaining other process parameters steady significantly increases MOE (Lehmann 1970).

Heebink et al. (1972) attempted to measure the vertical density gradient and MOE for particleboard with core moisture of 5 % and surface moisture 15 %. The high density regions (surface layers) resulted in high effective MOE values. Plath (1971b) also pointed out the optimum gradient for maximum MOE is a relatively high density surface. This indicates that bending strength is mainly dependent on the surface of a beam element.

The modulus of elasticity is strongly dependent upon flake length. Heebink and Hann (1959) found that longer flakes produce particleboard with substantially higher effective MOE values. However, the effect of particle thickness on MOE does not appear to be significant. Gatchel et al. (1966) found an increase in MOE when flake thickness decreased from 0.03 to 0.015 inch.

3.4.3.2 Internal Bond

The internal bond (IB) strength of particleboard is defined as the tensile strength perpendicular to the panel's plane. A well-cured particleboard will normally fail when stressed in tension perpendicular to the middle of the board which is the lowest density region of the board. The weakest particle interaction and lowest consolidation is found in this region.

It has been reported that highly densified surfaces increase the bending strength of particleboard, but the resultant lower density core region normally reduces the internal bond (Strickler 1959). The lower density core is a result of a lower press cycle, which causes excessive moisture in the core. The trapping of the excessive moisture leads to spring-back of the board, breaking inter-particle bonds and creating a lower density core. Therefore, IB will be reduced with the increase of surface moisture as the excessive moisture is trapped after hot pressing.

Talbott and Maloney (1957) suggested that the internal bond strength improves as the core particle configuration changes from a long wide flake to planer shavings or silvers. A three-layer particleboard, with coarse particles in the core and flakes at the surface, is often used to optimize board properties (Suchsland 1960). Further research has also found that IB improves with increasing amounts of wood dust (particles < 0.2 mm) up to 20 % and then starts decreasing (Nemli et al. 2006). Smaller particles increase the contact between blended materials, filling the gaps inside the core and increasing the resistance to tension perpendicular to the surface.

Internal bond is known to be influenced by the wood species used to produce particleboard. Internal bond strengths decrease with decrease in wood species density (Ahsori and Nourbakhsh 2008). Loh et al. (2010) investigated the physical and mechanical properties of particleboard from admixtures of rubberwood and Mahang at different proportion levels. The researchers reported that increasing quantities of low-density wood particles in the mix causes a significant reduction of internal bond strength.

Several investigations of the effects of adding wax during particleboard production on the IB strength of particleboard have been conducted. Some studies have reported significant improvement in IB by adding small portions of wax (Talbott et al. 1957; Hann et al. 1962). However, Heebink and Hann (1959) observed no changes to IB strength after adding 1% wax

into UF-bonded particleboard. Due to the limited information provided, it is not possible to confirm the relationship between wax and IB strength.

3.5 Wood Parameters

Particleboard is commonly made of various raw materials on a dry weight basis. Therefore, the properties of the raw material have a significant effect on both the manufacture and the physical properties of the final product. The quality of wood furnish in particleboard is controlled by the species properties such as density, acidity, extractive content and machinability.

In the particleboard industry, a great variety of wood species are used as raw materials. The choice of the wood species used is often determined by the availability and price of the raw material.

Particle sizes and shapes used in particleboard vary widely, and particle size plays an important role in affecting the properties of particleboard. The influence of particle size and shape on mechanical properties of boards is well described in several papers in the research literature. The central statement of these papers is an increase of bending strength, and compression and tension strength in the board plane, but a decrease of internal bond strength with increasing particle length. Thus, in the particleboard industry, it is important to ensure the overlapping areas of particles are large enough to guarantee the transmission of the wood strength to the strength of the whole assembly.

The effect of wood species density on particleboard density is interdependent. Species of lower density than final particleboard density are required to produce high quality particleboard economically (Vital et al. 1974; Hse 1975). The final board density should be higher than the initial wood species density in order to attain better inter-particle contact and hence sufficient bond between particles by maximizing the usage of resin. Otherwise, most of the resin will polymerize at the void spaces resulting in poor inter-particle bonding. Therefore, the wood furnish needs to be compressed to a higher specific gravity than their original to obtain sufficient inter-particle contact and thus improve resin efficiency.

Haygreen and Gartjejansen (1971) made flake-type particleboard at density of 721 kg/m^3 by using five tropical hardwoods with medium to low density (Aceituna, Banak, Jogo, Gallina and Aspen). UF resin was used for this investigation. It was found that the boards made from the species with lower densities as furnish could attain better compaction and inter-particle contact than higher density species when producing particleboard at the same normal density. This results in superior bending properties in boards made with lower density species.

Vital et al. (1974) produced particleboard from four exotic hardwood species with different densities. Boards were made with the mixtures of all wood species at each of two compaction ratios (board density:species density), 1.2 and 1.6. They observed that the board pressed with a higher compression ratio has a higher bending strength than the board with a lower compression ratio. This was attributed to the greater volume of wood compacted and the increased inter-particle contact, which, combined, produced better bonding.

Xu and Suchsland (1999) investigated the effect of mixed species on particleboard properties, and reported that the bending properties of mixed species particleboard were less than the single species counterpart. The major disadvantages of mixed species particleboard may relate to the variation of density that affects the resin uptake by particles of different species. During mixing, lighter density particles have a tendency to flow on top of the mixing tank and hence absorb more resin than the heavy particles. This causes the poor adhesion interference between the heavy and lighter particles after they are formed into particleboard.

Acidity of the species is also important in determining the suitability of species for particleboard. Different wood species can show great differences in pH. A chemical-induced effect can occur if the wood species have acidic behavior, which might cause acceleration of the hardening of adhesives based on polycondensation resins. Particleboard produced with urea-formaldehyde resin favours acidic conditions for faster resin curing. Therefore, the acidity of the furnish should be monitored to allow proper adjustment of the catalyst level to prevent long press cycles or pre-cure of the mat before reaching sufficient consolidation.

The material from which high quality particleboard can be economically produced is limited to those materials of adequate inherent strength which can be readily broken down and glued together without excessively destroying the native strength. Cost, as influenced by resin content, is one of the concerns, as many materials could theoretically be used for

particleboard, but the higher adhesive content required to obtain a satisfactory board makes them too expensive.

3.6 Processing Conditions and Parameters

3.6.1 Resin Efficiency

As stated above, resin consumption is one of the major expenses for the particleboard manufacturer. Therefore, an optimum board property with minimum adhesive consumption is the goal of all manufacturers, and is known as resin efficiency.

Resin efficiency in particleboard is difficult to study directly due to the problems encountered in quantitatively measuring the amount of liquid adhesive sprayed on the surface of irregularly-shaped particles. Therefore, the resin distribution onto the particles has to be examined. Klauditz and Meinecke (1962) suggested applying the resin to the particles by spraying fine droplets of the resin onto continuously agitating particles. This agitation allows some interaction between the particles to further disperse the adhesive. Several studies found that resin efficiency increases as the droplet size from the spray nozzle decreases and the degree of dispersion of the adhesive increases (Carroll and Mcvey 1962; Meinecke and Klauditz 1962; Lehmann 1970). An investigation was carried out by Lehmann (1970) to determine the relative area coverage on particles with three different resin contents (2 %, 4 % and 8 %) and two atomization level (fine and coarse). A dye was added to the resin for investigation. By measuring the reflection of the dyed resin, it was found that fine atomization results in better particle coverage as each particle will be uniformly covered with the finer adhesive spray.

In addition to adhesive droplet size, the interaction of particles is also important for resin efficiency. It has been found (Myron, 1977) that increasing the rubbing action between individual particles as they are agitated in the mixer enhances the possibility for resin transfer from one particle with excessive adhesive to another particle deficient in adhesive. Christensen and Robitschek (1974) found that the extended post-blending agitation of furnish produces higher internal bond values a compared to boards from furnish without post-blending.

In relation to particle geometry, Klauditz (1962) states: “The thinner the chips and – consequently – the greater the total surface to be coated, the greater care to be given to thorough blending.” Lehmann (1970) observed that smaller particles always have larger percentage surface area coverage than coarser particles when the two are blended together, regardless of whether fine or coarse spray is used. Also, lstrate et al. (1964) used a slenderness coefficient to describe the particle shape. Particles with high slenderness coefficients (length/thickness) could be satisfactorily glued into particleboards without requiring a continuous layer of adhesive. Thicker particles had less surface area per unit of weight but required more adhesive than did thinner particles. The importance of particle surface area and its relation to resin distribution also has been reported by Carroll and McVey (1962) and Maloney (1970).

3.6.2 Mat Moisture Content

The mat moisture content is recognized as an important medium for the heat transfer from surface to the core layer during hot pressing which assists the resin curing of particleboard. However, excessive moisture migration to the core imposes the requirement of excessively long press cycles to allow removal of moisture through the edges to prevent delamination of the board upon pressure release. Excessive moisture may cause rapid densification of the surface and loose core, resulting in poor internal bond strength and poor screw withdrawal strength on the final board.

In order to overcome these adverse moisture effects, non-uniform moisture distribution is utilized through the mat thickness. Normally, a non-uniform moisture distribution, with the surface layer at higher moisture content than the core, accelerates the heat transfer without lengthening the press cycle. Heebink (1974) obtained an improvement of MOR and MOE values by applying the non-uniform moisture distribution (15 % in surface and 5 % in core) in particleboard.

Strickler (1959) found that the pressing time for heating the mat core decreased with increasing surface moisture content. However, with the continuous increase in surface moisture above its optimum levels, the MOR, MOE and IB values started reducing. According to Strickler (1959), when the surface moisture increases above 15 % with core moisture at 9 %, the strength properties decline. Lehmann (1960) also found a reduction of

MOR and IB values of particleboard with a further increase in surface moisture from 16.5 % to 20 %.

Suchsland et al. (1974) also found that the release of pressure when the moisture content in the middle is higher may lead to spring-back or non-reversible excessive dimensional changes. Therefore, stress relief or the opening of the press after hot pressing should be addressed.

3.6.3 Pressing Time and Temperature

Pressing time is the total time taken from when the upper platen first touches the wood mat until it leaves it. Pressing time should be sufficient to consolidate the particle mat into the desired thickness as well as the polymerization of resin into cross-linked solid polymer to hold the mat in a compacted form after removal from the press.

Pressing time and temperature are extremely important parameters in particleboard manufacturing and have to be carefully controlled to ensure that the core temperature attains the level required to cure the adhesive without subjecting the board surface to a high, degradative temperature. Because the entire board is not uniformly heated throughout the thickness, the curing of the adhesive does not occur uniformly; the adhesive at the mat surface is the first to cure and that at the central region the last. Since the mat centre is always at the lowest temperature, the pressing time and temperature should be adequate to ensure that the core reaches a sufficiently high temperature to allow the resin to cure. This can be achieved by increasing the press time at a constant temperature, or by increasing the press temperature at a constant pressing time.

Lehmann et al. (1973) found that shorter pressing times are obtained with a higher pressing temperature. This higher temperature allows faster heat transfer and shifts the maximum density region toward the core, resulting in an increase in internal bond strength and a reduction in the bending strength decrease. However, Lynam (1959) stated that a longer press cycle at lower temperature was desirable. He believed that at this condition, less water was evaporated and the remaining water was more uniformly distributed in the finished board.

Resin type is one of the factors affecting the resin curing process. Lehmann et al. (1973), reported that phenol-formaldehyde resins have a slower curing rate than urea-formaldehyde resins. Therefore, the selection of the appropriate resin type is important in order to attain short pressing time.

3.6.4 Resin or Adhesives

Resin plays an important role in particleboard production as it works as a binder for the particles to bond together. A number of different types of resins are used in the particleboard industry, and each has its advantages. However, the effect of resin on production costs is the main concern, as resin costs are a major portion of the total manufacturing expense of particleboard.

Temperature is an important parameter controlling resin reactivity in particleboard production, as it directly relates to resin curing. Press temperature must be limited to prevent board surfaces in contact with the platens from being degraded; therefore, increasing the core temperature is normally accomplished by lengthening the press cycle. Resin with a lower reactivity might require a higher temperature and longer press cycle (Carroll 1963; Lehmann et al. 1973). Therefore, it can be concluded that resin type has a significant effect on the press cycle and temperature.

The adhesive level used can also affect particleboard properties. Kimoto et al. (1964) made particleboard using urea-formaldehyde resin at levels of 8 %, 10 % and 15 %, and found only slight improvement in both strength and thickness swelling properties with 15 % as compared to 10 % adhesive. Lehmann (1970) used a urea-formaldehyde adhesive at 2 %, 4 % and 8 % solids and also found only small increases in MOR, MOE and thickness swelling when adhesive content increased from 4 % to 8 %, but internal bond continued to increase substantially. These results indicate that increasing resin content in particleboard does not significantly increase the strength properties of particleboard.

The durability of particleboard may be improved with an increase in the resin level. Hann et al. (1963) reported improvement of particleboard durability when the adhesive level was increased from 3 % to 6 % and 4 % to 8 % for phenol-formaldehyde and urea-formaldehyde

respectively. Lehmann (1974) also reported much lower spring-back after accelerated ageing when the phenol-formaldehyde adhesive content was increased from 3 % to 9 %.

There are no consistent data available which indicate a particular adhesive level is optimum. Basically, adhesive contents are based on the oven-dry wood weight, which is extremely dependent upon particle configuration. Each particle configuration will have an optimum adhesive content dependent upon the desired board and the economics of production.

Several investigations have used dipping particles into aqueous solutions of resin before being sprayed with conventional resin solution. This method, called impregnation of resin, may improve particleboard properties (Browne et al. 1966; Haygreen and Gertjejansen 1971). Significant improvement of spring-back and thickness swelling were observed, as well as board properties. However, the cost of producing this impregnating material limits the applicability.

3.6.4.1 Resin Type

There have been a number of studies of particleboard production using different types of resin. Urea-formaldehyde (UF), melamine formaldehyde (MF) and phenol-formaldehyde (PF) are the most commonly used resins in the particleboard industry.

Melamine-Formaldehyde (MF)

Melamine-formaldehyde resin is a synthetic resins obtained by the chemical combination of melamine (a crystalline solid derived from urea) and formaldehyde (a highly reactive gas obtained from methane). It is a complex, interlinked polymer that cures to a clear, hard and chemically-resistant resin after heating. Melamine formaldehyde is commonly used as a particleboard and plywood adhesive.

MF is stain-resistant and resistant to strong solvents and water. Also, it is white, tasteless, odourless and exhibits great chemical and heat resistance. Unlike other types of thermoplastics that soften with heat and harden when cooled, MF retains its strength and shape once it is cured.

Melamine formaldehyde is affected by alkalis and concentrated acids such as sulphuric acid (H_2SO_4) and oxalic acid ($H2C_2O_4$). Melamine formaldehyde utensils are not microwave-safe.

They absorb radiation, which causes their polymer bonds to break down and percolate toxins into the food. Melamine formaldehyde ingestion leads to kidney failure. MF is relatively more expensive than UF and PF.

Phenol Formaldehyde (PF)

Phenol formaldehyde, also known as phenolic resin, is made by reacting phenol (an aromatic alcohol derived from benzene) with formaldehyde (a reactive gas derived from methane). PF resin was the first completely synthetic polymer to be commercialized.

PF resin is resistant to cold curing and all common organic solvents. However, it has a lower resistance to strong alkalis. Although PF resin is comparatively brittle, this is not a significant property for many applications. Its dark colour is often a disadvantage and the addition of a white pigment decreases it to a limited extent only. The durability of PF resin varies with different timbers, but factors such as glueline thickness can also have an effect.

PF resin is considered tolerant to most fungicidal chemicals, e.g sodium pentachlorophenate and isosafrole. In gluing wood, the moisture content of the timber is generally a more important factor than resin content. In this respect, greater latitude is claimed by dispersing, in normal alkali-condensed glue, a powdered phenolic resin condensate that is insoluble at room temperature but capable of swelling as the pressing temperature is reached.

In recent years, the curing temperature of the PF bonded plywood has come down from the region of 130 – 140 ^{o}C to 105 – 115^{o}C. This is an importance decrease and has justified PF resin being classified separately as a medium-temperature plywood glue.

Urea Formaldehyde (UF)

Urea Formaldehyde (UF) is a cost-effective non-transparent thermosetting resin that is made from urea and formaldehyde heated in the presence of a mild base such as ammonia.

UF is the most important amino-plastic resin in the wood-working industry due to its high reactivity, water solubility and the reversibility of the amino-methylene link, which also explains the low resistance of UF resins to the influence of water and moisture, especially at higher temperatures.

During the curing process, UF resins forms into three-dimensional networks that are no longer thermo-formable and insoluble. The curing of the UF resin has to be done in an acidic environment. This indicates that better curing can be reached by decreasing the pH of resin.

This acidic condition can be achieved by the addition of direct acid or latent hardener. Ammonium sulphate or ammonium chloride is widely used as a latent acid in the particleboard industry. The hardener reacts with the free formaldehyde in the resins to generate acid. The portion of hardener to be used depends on its ability to release acid. The speed of reaction depends on the amount of available free formaldehyde and hardener, which is dependent on the temperature and time. Pizzi (1983) found that NH_4Cl (a weak acid) is a better hardener than HCl (strong acid), as the latter produces weaker inter-particle bond.

UF resin is not stable at higher relative humidities especially at elevated temperatures, since the amino-methylene linkage is susceptible to hydrolysis (Yamagushi et al. 1980). Also, Conner (1996) found that UF-bonded board lacks resistance to moisture, especially in combination with heat, compared to PF- and MDI-bonded board. Melamine has been suggested to be added into UF resin to improve its resistance to moisture or humidity (Dunky 1998). However, this might change its properties and also the cost of the resin.

3.6.5 Previous Work on Resin used in Particleboard

A number of investigations have been made in order to discover the most suitable resin for the particleboard industry.

Deppe and Ernst (1971) produced particleboards with di-isocyanate binders and phenol formaldehyde respectively. It was found that press times for di-isocyanate are much shorter than the relatively long times for phenol formaldehyde but the di-isocyanate-bonded material stuck to cauls and other metal surfaces during manufacture.

Kojima et al. (2010) completed an investigation of particleboard using hinoki particles and different amounts of MDI resin (2 %, 4 %, 6 % and 9 %) and PF resin (6 % and 10.2 %). The resin content equivalence for the two types of resin was determined based on internal bond (IB) strength. It was found that the MDI board required only 50 % to 70 % of the resin required for a PF board with a comparable IB quality. MDI-bonded board more superior in performance in all tests, with the exception of the boiling treatment, in which no significant difference was observed between MDI and PF.

Papadopoulos and Hill (2001) compared the properties of board made from MDI and UF resin, and reported that MDI-bonded boards have superior properties to UF- bonded boards. It was found that the amount of MDI resin required to produce a board with the same properties is relatively lower compared to that of the UF resin. Although the mat moisture content and temperature have a significant influence on the bonding efficiency in UF-bonded boards, they do not have any significant effect on the bonding efficiency in MDI boards.

Dunky (1998) suggested that weather durability can be increased by the addition of hydrophobic chains into the hardened network. This can be achieved by incorporating urea-capped di- and tri-functional amines which contain aliphatic chains into the resin structure, and using the hydrochloride derivates of some of these amines as a curing agent (Ebewele et al. 1991).

The amount of hardener should be maintained at optimal level to create natural glue-lines which show a distinctly improved resistance to hydrolysis. Adding excessive hardener to the resin does not increase the curing reaction. It will leave residues of acids in the glue-lines and contribute to the brittleness of cured resin. This will commence the hydrolysis of the wood cell wall adjacent to glue-lines as well as acid-catalysted resin degradation, which decreases board durability (Myer 1984).

3.7 Particleboards from different types of raw materials

Evergreen Mediterranean hardwoods as particleboard raw material

The wood and biomass (branches and foliage) of five Mediterranean evergreen hardwood species (*Quercus coccifera, Quercus ilex, Arbutus unedo, Phillyrea latifolia, Erica arborea*) growing in Greece have been investigated as particleboard raw material. The average diameter of the stems varied between the species from 4.1 cm (*Erica arborea*) to 7.5 cm (*Quercus ilex*) and the bark-to-wood ratio of the stems varied from 7.8 % (*Quercus ilex*) to 15.9 % (*Erica arborea*).

The bulk density of particleboard furnishes produced by processing the above materials through a chipper and a ring flaker varied from 143.6 to 162.5 kg/m^3. The common industrial

furnish used in laboratory conditions has a bulk density of 143 kg/m^3. Particle size and their distribution also varied between the hardwood species. One-layer laboratory particleboards made with the above materials at densities of 650, 700 and 750 kg/m^3 had considerable lower mechanical properties but better dimensional stability than boards made with common industrial furnish. All properties were affected by wood species and board density. The internal bond of all boards was higher than the value required by EN 319/1993 but adequate bending strength at moderate densities (0.70 kg/m^3) was achieved only in boards made with *Arbutus unedo* and *Quercus ilex.* Boards made with branches and foliage had very low mechanical properties.

Properties of Particleboard Produced from mixtures of Rubberwood and Mahang Species

The objective of this study (Loh et. al, 2010) was to investigate the physical and mechanical properties of particleboard produced from admixtures of rubberwood and Mahang at different proportion levels and the properties were evaluated based on the Japanese Industrial Standard for particleboard (JIS A 5908-1994). The physical and mechanical data presented here, were analyzed using Analysis of Variance (ANOVA) and Least Significant Difference (LSD), to determine the significant differences of the variables.

From the study, an increase in density was observed to result in an increase of thickness swelling of particleboard. In addition, the water absorption for particleboard with density of 500 kg m^{-3} was higher. Particleboards of rubberwood and Mahang had significantly lower wet and dry bending properties. In terms of internal bond strength, a decrease in rubberwood proportion resulted in a decrease in the internal bonding strength of the particleboard.

Producing Particleboard Using Mixtures of Bagasse and Industrial Wood Particles

In this study (Tabarsa, 2011) the feasibility of using of mixtures of bagasse and industrial wood particles for producing single- and three-layer particleboard is explored. The aim of this study was to consider bagasse as a partial substitute raw material for the particleboard industry. Variables were type of board at two levels (single- and three-layer), percentage of bagasse added to industrial particles at 4 levels (20 %,30 %, 40 % and 50 %) , and press temperature at two levels (165^{o}C and 180 ^{o}C). In producing three- layer particleboard, wood and bagasse particles were separated and placed in different layers so that bagasse particles were located in the surface layers and wood particles were placed in the core of board.

However, in one layer particleboard bagasse and wood particles were used as a mixture. The efect of variables on the physical and mechanical properties of particleboard were determined, and the results showed that in three-layer particleboard physical and mechanical properties were better than single-layer particleboard. Increasing press temperature caused improvement in particleboard properties in most cases due to intermeshing and increasing softening wood and bagasse particles. The optimum treatment in this study was found to be adding 50 % bagasse and a press temperature of 180 ^{o}C.

Producing Particleboard Using Sunflower Stalks (helianthus annuus l.) and Poplar Wood (populus alba L.)

In this study, (Bektas et. al, 2005) three-layer particleboards were produced from a mixture of sunflower stalks (Helianthus annuus L.) and poplar wood (Populus alba L.) at certain ratios utilizing urea-formaldehyde (UF) adhesives. Panels with a density of 0.7 g/cm ^{3}were manufactured with the ratios of 25 %, 50 %, and 75 % particles from sunflower stalks or poplar. For comparison, panels were manufactured solely with sunflower stalks and poplar wood.

All panels were tested for physical properties (thickness swelling (TS) and water absorption (WA)) and the mechanical properties (internal bond (IB), modulus of elasticity (MOE), modulus of rupture (MOR), and screw-holding capacity (SHC)). The results showed that all the panels provided the properties required by the Turkish standards for general purpose particleboards. Furthermore, the properties of the panels improved with the rising percentage of poplar particles in the panels.

The combination of sunflower stalks and poplar particles can be used at different ratios and the resulting panels can be utilized in indoor applications for general purposes such as furniture manufacturing. This result indicates that sunflower stalks can be an alternative raw material for the manufacture of particleboards.

Producing Particleboard Using Hardwood Sawmill Residues

Hardwood sawmill residues have traditionally not been favoured for the particleboard industry due to their high density and extractive content (Nirdosha, 2007). However, recent re-growth and plantation of timber have produced hardwood sawmill residues with lower density and extractive contents. This study (Nirdosha, 2007) investigated the use of hardwood sawmill residues in producing particleboard. An experimental design covering seven

variables with two levels was performed to manufacture three-layer particleboards in the laboratory. Resin surface and pressing time were found to be significant factors that influence the mechanical and physical properties of the final boards. The need for higher moisture content and resin load compared to softwood particleboard needs to be addressed further to achieve an economically-feasible board.

Particleboard from Agriculture Residues

Agriculture residues such as wheat straw and flax fibre have become popular alternative raw materials for particleboard production. Mo et al. (2003) investigated the potential of different resin types as a binder for wheat-straw particleboard. They found that MDI resin is the most suitable resin for wheat-straw particleboard as MDI facilitates working with high moisture content better than other resin types. This resin can effectively wet the surface of the straw, enhancing proper chemical bonding through both hydrogen bonding and covalent bonding (Mo et al. 2003). Wood-based resin such as UF resin cannot effectively wet straw surface due to the hydrophobic wax and silica found on the surface of the straw (Hague et al. 1998).

Flax fibre has also been a popular raw material which has recently been used not only in particleboard production but also in other composite industries due to its low density, high specific stiffness, recyclability and low cost (Troger et al. 1998). It has been identified as a potential alternative lignocellulose material which could supplement wood for particleboard production. Some researchers (Papadopoulos and Hague 2003) have investigated the possibility of using flax particles by partially substituting flax shive with wood chips bonded with UF resin to make particleboard. According to the report, 30 % substitution of flax could produce particleboard with the strength and physical properties required by the industry standard for interior use.

Ergun et al. (2008) investigated the feasiblity of pepper stalks in the production of particleboard. Particleboard with density of 0.53, 0.63 and 0.73 g cm^{-3} were manufactured from pepper stalks using certain ratios of urea formaldehyde (UF) and melamine urea formaldehyde (MUF) resins. It was found that pepper stalks have higher cellulose content than hardwoods and softwoods. The amount of extractive and lignin content is significantly lower than hardwoods and softwoods. Basically, pepper stalks' fibre properties are similar to hardwood fibres. The results of the study demonstrate that pepper stalks may be an alternative raw material for particleboard industry.

3.8 Australian Standard for Particleboard

The Australian Standard for particleboard was first published in February 1997. The code of Australian Standard for particleboard is AS/NZS 4266:2004. In this standard, particleboards are classified into following categories:

- Standard particleboard
- High moisture-resistant (HMR) particleboard
- Particleboard flooring
- Treated products

These particleboards have applications in different areas. Standard particleboard is normally used in dry conditions such as furniture or for interior structures, while high moisture-resistance particleboard is applicable for use in interior areas under humid conditions where the likelihood of wetting is very high, e.g. kitchen bench tops, bathrooms. Materials such as wood veneer, vinyl, melamine are usually used in laminating these HMR boards.

Particleboard flooring has more stringent bending strength and stiffness requirements in the Australian Standard. Particleboard flooring is divided into two classes. Class 1 flooring is for the condition where the ambient temperature is above 25^{o}C and the environment is humid, which may occur during construction; the regions where resistance to extreme dampness is not necessary are classified as Class 2 flooring.Treated particleboards are normally treated to protect them against certain potentially destructive organisms or elements. There are three types of treatments fungicide , termiticide, and fire-retardant.

Table 3.1 below shows the typical or target property values for AS/NZS 4266:2004 Standard Particleboard.

Property	**Units**	**Thickness Class - mm**
		13 - 22
Density	kg/m^3	660 - 680
Bending Strength (MOR)	MPa	15
Bending Stiffness (MOE)	MPa	2600
Internal Bond Strength (IB)	kPa	450

Table 3.1 Typical or target property values for Standard Particleboard

AS/NZS 4266:2004 provides guidance on the testing of quality of reconstituted wood-based panels. This standard provides a list of methods as shown in Table 3.2 below.

AS/NZS Number	**Title**
4266.1	Method 1: Sampling, cutting, and conditioning of test pieces
4266.2	Method 2: Dimensions, squareness, flatness, and edge straightness of whole panel
4266.3	Method 3: Moisture content
4266.4	Method 4: Density
4266.5	Method 5: Modulus of elasticity in bending and bending strength
4266.6	Method 6: Tensile strength perpendicular to the plane of the pnel (internal bond strength)
4266.7	Method 7: Surface soundness
4266.8	Method 8: Swelling in thickness after immersion in water
4266.9	Method 9: Thickness stability and glue bond quality
4266.10	Method 10: Wet bending strength after immersion in water at 70^{o}C or boiling temperature
4266.11	Method 11: Moisture resistance under cyclic test conditions
4266.12	Method 12: Surface water absorption
4266.13	Method 13: Resistance to axial withdrawal of screws
4266.14	Method 14: Dimensional changes associated with changes in relative himidity
4266.15	To be withdrawn
4266.16	Method 16: Formaldehyde emission – Desiccator method
4266.17	To be withdrawn
4266.18	Method 18: Efficacy of fungicide treatment
4266.19	Method 19: Assessing appearance of melamine overlaid panels

4266.20	Method 20: Resistance to surface abrasion (Taber abrasion test)
4266.21	Method 21: Over-cure or under-cure of resin
4266.22	Method 22: Porosity of laminated surface
4266.23	Method 23: Resistance to steam
4266.24	Method 24: Resistance to cracking
4266.25	Method 25: Resistance to staining
4266.26	Method 26: Resistance to dry heat
4266.27	Method 27: Resistance to impact
4266.28	Method 28: Bond strength of PVC films
4266.29	Method 29: Heat resistance of PVC films
4266.30	Method 30: Moisture resistance of finished foils
4266.31	Method 31: Adhesion of paper foils by the crosscut test
4266.32	Method 32: Veneer bond strength
4266.33	Method 33: Veneer bond durability
4266.34	Method 34: Surface absorption
4266.35	Method 35: Dimensions of test pieces

Table 3.2 List of methods of AS/NZS 4266:2004

3.8 Summary

The review of literature has identified the basic process of particleboard manufacture, the processing parameters which affect the board properties and the basic mechanical properties required for general purpose particleboard. Following are the processing parameters identified:

- Surface moisture content
- Core moisture content
- Surface resin content
- Core resin content
- Pressing time
- Pressing temperature
- Particle size distribution

The mechanical properties required to meet the specifications of the AS/NZS Standard are:

- Modulus of Rupture (MOR)
- Modulus of Elasticity (MOE)
- Internal Bond (IB)

The typical ranges of properties required in a general purpose particleboard have also been identified as below:

- Modulus of Rupture (MOR) – 12 – 16 MPa
- Modulus of Elasticity (MOE) – 2400 – 2700 MPa
- Internal Bonding (IB) – 400 – 450 kPa

Chapter 4

DESIGN OF EXPERIMENTS (DOE)

4.1 Overview

Due to the large number of process variables observed to affect particleboard properties, the theory of experimental design was considered as a possible method of planning and analyzing experiments. Many previous researchers have applied DOE (Design of Experiments) to plan and execute research involving a large number of variables.

4.2 Introduction

Test procedures in scientific and engineering experiments are often primarily guided by traditional laboratory procedure and subjective considerations of practicality. Such experimental procedures are often viewed as economical in terms of the number of runs that must be conducted during the study period. However, the economy of attempts can be deceiving for two reasons. First, economy is often achieved by severely limiting the number of factors whose effects are studied. Second, the sequence of tests may require that only one of the factors of interest be varied at a time, thereby preventing the evaluation of any joint effects of the experimental factors. The effective use of statistical principles in the design of experiments ensures that experiments are designed economically, that they are efficient, and that individual and joint factor effects can be evaluated.

Design of experiment (DOE) theory is a subset of statistics which provides the experimenter with methods for selecting the values for independent variables, so that a limited number of experiments can be performed to obtain an economical laboratory protocol. DOE is also a very important technique when there is a need to develop a new product using new materials, if the underlying mechanism in the system to formulate a model between response variables and independent factors is unknown. This theory is suitable for the primary objective of our research: to develop a technology for producing an economical particleboard utilizing large quantities of hardwood sawmill residues, meeting the AS/NZS 1859.1:1997.

This chapter therefore covers a brief introduction to DOE and discusses methods of DOE and analysis used in the research.

4.3 Fundamentals of DOE

An experimental design is a procedure that enables an experimenter to perform scientifically sound experiments with the aim of drawing certain conclusions about the situation being studied. Typically, experimental subjects (or units) are subjected of the treatments after the experiment has been concluded. The data gathered are then analysed, either graphically by using suitable plots, or by means of statistical tests. Indeed, DOE is essential in any field that attempts to obtain information on a process and to use this information to optimize and improve production.

A well-designed experiment should be able to:

1. Provide answers to conjectures about the response to different treatments
2. Provide estimates of treatment effects and their precision, i.e. specify a range of values within which the true effects will fall with a certain prescribed probability
3. Recognize and eliminate extraneous variations from the comparisons under consideration.
4. Provide conclusions which are robust

4.3.1 Important Terminology

The terminology of experimental design is not uniform across disciplines or even, in some instances, across textbooks within a discipline. For this reason, a discussion of statistical experimental design with a brief definition of terms is provided in this section.

(a) **Factor**: A controllable experimental variable that is thought to influence the response, e.g. temperature, pressure, moisture content, etc. Each factor is either a treatment factor or a blocking factor, and it is either fixed or random.

(b) **Response**: Outcome or result of an experiment.

(c) **Level of factor**: Each factor is usually set at several levels, i.e. it is given several values. If the set of levels is numerical, the corresponding factor is referred to as being quantitative. The different combinations of factor levels provide the different conditions to which the experimental units are subjected. The response of the units to these varying conditions is subsequently analysed.

(d) **Randomization**: Normally, the order in which the experimental units are allocated to the different combinations of factor levels should be done in a completely random fashion. This is usually achieved by using a table of random numbers, or by tossing a die, or shuffling cards. The main aims of randomization are to reduce bias in the conduct of experiments and to ensure that measurement errors are independent.

(e) **Replication**: Repetition of an entire experiment or a portion of an experiment under two or more sets of conditions. An average test result will be obtained for analysis in order to monitor and minimize any human error.

(f) **Block**: A group of experimental runs conducted under relatively homogeneous conditions. Although every measurement should be taken under consistent experimental conditions (other than those that are being varied as part of the experiment), this is not always possible. Blocks are used in experimental design and analysis to minimize bias and error variance due to nuisance factors.

(g) **Confounding**. One or more effects that cannot unambiguously be attributed to a single factor or interaction.

4.3.2 Classes of DOE Design

Experimental methods are being increasingly used in manufacturing to optimize the production process. However, different production processes might have different situations or needs, such as number of factors or type of factor. Hence, an appropriate type of DOE design needs to be selected in order to fit the desired process situation. Following is a list of some of the most common DOE types:

1. One-factor designs
2. General full factorial designs
3. Two-level full factorial designs
4. Two-level fractional factorial designs
5. Plackett-Burman designs
6. Taguchi Methods: Robust Design Experiments
7. Response surface method designs
8. Reliability DOE

In many cases, it is sufficient to consider the factors affecting the production process at two levels. For example, the temperature for a process may either be set at a higher or lower temperature, the moisture content level of core, resin level of core and surface layer of particleboard, etc. The experimenter may like to determine whether any of these changes affects the results of the production process. The most significant design to study those varying levels of factors is the full factorial design, that is, to try all possible combinations of settings. This would work well, except that the number of necessary runs in the experiment (observations) will increase geometrically. For the particleboard experimental design, five factors are recognized. The necessary number of runs in the full factorial experiment would be $2^5 = 32$ runs. Since each run may require time-consuming and costly setting and resetting of machinery, it is often not feasible to require so many different production runs. In these conditions, fractional factorial design is a very efficient design to keep the main effects being computed correctly.

4.4 $2^{(k-p)}$ Fractional Factorial Designs at 2 Levels

4.4.1 Generating the Design

In general, it will successively "use" the highest-order interactions to generate new factors. For the fractional factorial design of particleboard, 8 factors are recognized. Therefore, the following design that includes 8 factors but requires only 33 runs (observations) is generated via MINITAB 15.

Board no	Moisture core	Moisture Surface	Resin Core	Resin Surface	Hardwood portion in core	Hardwood portion in surface	Press Temperature	Press time
1	13	14	16	16	60	60	200	240
2	7	9	8	16	40	40	200	120
3	13	14	16	8	40	60	150	240
4	7	14	8	16	40	60	150	240
5	7	14	8	16	60	60	150	120
6	7	14	16	16	60	40	150	240
7	7	9	8	8	40	40	150	240
8	13	14	16	8	60	60	150	120
9	13	14	8	16	60	40	200	120
10	7	9	16	16	40	60	200	240
11	13	9	16	8	40	40	200	120
12	13	14	8	8	40	40	150	120
13	13	9	16	8	60	40	200	240
14	13	9	16	16	60	40	150	120
15	13	14	8	8	60	40	150	240
16	13	9	8	16	40	60	150	120
17	13	9	16	16	40	40	150	240
18	13	9	8	16	60	60	150	240
19	7	9	8	8	60	40	150	120
20	7	9	16	8	60	60	150	240
21	7	14	8	8	60	60	200	240
22	13	14	8	16	40	40	200	240
23	13	9	8	8	40	60	200	240
24	7	9	16	8	40	60	150	120
25	10	11.5	12	12	50	50	175	180
26	7	9	16	16	60	60	200	120
27	7	14	8	8	40	60	200	120
28	7	14	16	8	60	40	200	120
29	7	9	8	16	60	40	200	240
30	7	14	16	8	40	40	200	240
31	13	14	16	16	40	60	200	120
32	7	14	16	16	40	40	150	120
33	13	9	8	8	60	60	200	120

Table 4.1 Example of fractional factorial design of particleboard with 8 factors

Reading the design. The design displayed above should be interpreted as follows. Each column contains +1's or -1's to indicate the setting of the respective factor (high or low, respectively). Note that there are numerous options provided to display (and save) the design using notation other than to denote factor settings. For example, actual values of factors (e.g., *90 degrees* Celsius and *100 degrees* Celsius) or text labels (*Low* temperature, *High* temperature) may be used.

Randomizing the runs. Because many other things may change from production run to production run, it is always good practice to randomize the order in which the systematic runs of the designs are performed.

4.4.2 The Concept of Design Resolution

The design above is described as a $2^{(5-1)}$ design of *resolution* V. This means that you study overall $k = 5$ factors (the first number in parentheses); however, $p = 1$ of those factors (the second number in parentheses) were generated from the interactions of a full $2^{[(5-1) = 4]}$ factorial design. As a result, the design does not give full *resolution*; that is, there are certain interaction effects that are confounded with other effects. In general, a design of resolution *R* is one where no *l*-way interactions are confounded with any other interaction of order less than *R-l*.

4.4.3 Plackett-Burman (Hadamard Matrix) Designs for Screening

When one needs to screen a large number of factors to identify those that may be important (i.e., those that are related to the dependent variable of interest), one would like to employ a design that allows one to test the largest number of factor main effects with the least number of observations, that is to construct a resolution III design with as few runs as possible. One way to design such experiments is to confound all interactions with "new" main effects. Such designs are also sometimes called *saturated* designs, because all information in those designs is used to estimate the parameters, leaving no degrees of freedom to estimate the error term for the ANOVA. Because the added factors are created by equating (aliasing, see below), the "new" factors with the interactions of a full factorial design, these designs will always have 2^k runs (e.g., 4, 8, 16, 32, and so on). Plackett and Burman (1946) showed how full factorial design can be fractionalized in a different manner to yield saturated designs where the number of runs is a multiple of 4, rather than a power of 2. These designs are also sometimes called Hadamard matrix designs. Of course, it is not necessary to use all available factors in those designs, and it may sometimes be desirable to generate a saturated design for one more

factor than you are expecting to test. This will allow you to estimate the random error variability, and test for the statistical significance of the parameter estimates.

4.4.4 Calculation of Effects

An effect is defined as a change in the average response corresponding to a change in factor-level combinations or to a change in experimental conditions. The calculation of effects is facilitated by the introduction of algebraic notation for individual responses and averages of responses. This notation is helpful in clarifying the concepts of confounding and design resolution that will be discussed later in the chapter.

Factors are denoted in designed experiments by uppercase Latin letters: A, B, C, …, K. An individual response is represented by a lowercase Latin letter, usually *y*, with one or more subscripts. The subscripts designate the specific factor-level combination from which the response is obtained. There may also be one or more subscripts designating repeat observations for fixed factor levels. For example, the factors and their levels could be designated as below:

Factor	Factor Symbol	Level	Effect Representation	Subscript Symbol
Alloy	A	Steel	-1	*i = 1*
		Aluminium	+1	*i* = 2
Sleeve	B	Porous	-1	*j= 1*
		Nonporous	+1	*j= 2*
Lubricant	C	Lub 1	-1	*k= 1*
		Lub 2	+1	k= 2

Table 4.2 Example of factors and their levels

Effects for two level factors are normally categorized into three types as below:

- **Main effect:** The difference between the average responses at the two levels of a factor.
- **Two-factor interaction:** Half the difference between the main effects of one factor at the two levels of a second factor.
- **Three-factor interaction:** Half the difference between the two-factor interaction effects at the two levels of a third factor.

From the definition of a main effect as the difference in average responses at each level of a factor, the main effect can simply be calculated as Equation 4.1

$A = Y_{A+} - Y_{A-}$ Equation 4.1

The example shown in Table 4.2 simply illustrates a better understanding of the effect calculation. Table 4.3 below shows the effect representation and the calculated effects.

Effect Representation

A	B	C	AB	AC	BC	ABC	Response
-1	-1	-1	+1	+1	+1	-1	y_{111}= 60
-1	-1	+1	+1	-1	-1	+1	y_{112}= 52
-1	+1	-1	-1	+1	-1	+1	y_{121}= 54
-1	+1	+1	-1	-1	+1	-1	y_{122}= 45
+1	-1	-1	-1	-1	+1	+1	y_{211}= 72
+1	-1	+1	-1	+1	-1	-1	y_{212}= 83
+1	+1	-1	+1	-1	-1	-1	y_{221}= 68
+1	+1	+1	+1	+1	+1	+1	y_{222}= 80
Calculated effects							
23	-5	1.5	1.5	10	0	0.5	

Table 4.3 Example of effect calculation

The elements in the interaction column AB are the products of the individual elements in the columns labelled A and B. For example, the third element in the AB column is the product of the third elements in the A and B columns: (−1)(+1) = −1.

Calculated effect A:

$A = Y_{A+} - Y_{A-}$

$$A = \frac{(72+83+68+80)}{4} - \frac{(60+52+54+45)}{4}$$

$A = 23$

Calculated effect B:

$B = Y_{B+} - Y_{B-}$

$$B = \frac{(54+45+68+80)}{4} - \frac{(60+52+72+83)}{4}$$

$B = -5$

Calculated effect of AB:

$AB = Y_{AB+} - Y_{AB-}$

$$AB = \frac{(60+52+68+80) - (54+45+72+83)}{4}$$

$AB = 1.5$

The average effect on the response value is important for analysing experimental results as the higher the value, the greater the effect on the response. Therefore, this can be used to identify the most important factors on the response as well as to screen out the least important factors. Then, the analysis of variance (ANOVA) is generally used to confirm this. The sign of the calculated value of the effect determines whether the factor has a positive or a negative effect on the response. If the sign is '+', then the factor has positive effect on the response, otherwise the factor has a negative effect on the response.

4.4.5 Aliases of Interaction: Design Generator

Effects that cannot be estimated separately from one another are said to be confounded. Confounding occurs when a fractional factorial design is used, because all factor level combinations are not run. For example, if factor A is confounded with the 3-way interaction BCD, then the estimated effect for A is the sum of the effect of A and the effect of BCD. These effects are also said to be aliased. The alias structure describes the confounding that occurs in the design.

Effects that are aliased, or confounded, cannot be estimated separately from one another. For example, if the two-way interaction, BC, is confounded with the three-way interaction, ADE, it will not be possible to tell whether a significant effect is due to the BC interaction or the ADE interaction.

The key to the alias structure is the identity statement, for example, I + ABCDE. To determine which effects are confounded, multiply the term of interest by the identity statement and then eliminate the squared terms. For example, to find the term that BC is confounded with:

$$(BC)(I + ABCDE) = BC + AB^2C^2DE = BC + ADE$$

Therefore, BC and ADE are confounded with one another.

Considering a manufacturing plant with three factors, each at two levels, suppose eight test runs are to be made. However, due to the expense involved, only four test runs could be conducted. The cube plot in Figure 4.1 shows that only the first four test runs are conducted.

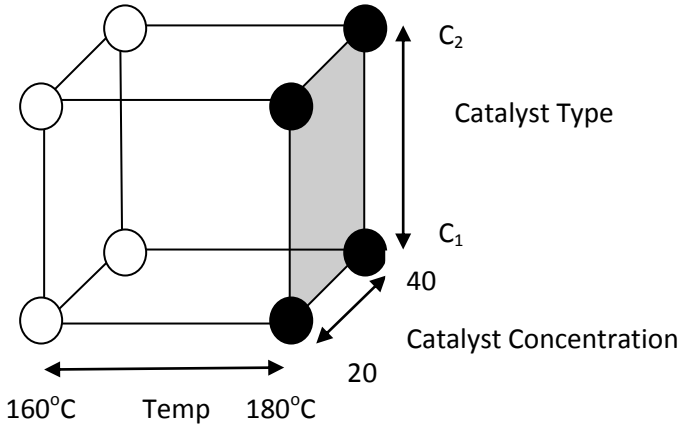


Figure 4.1 Cube plot demonstrates fractional factorial design

The effects representations for the various experimental effects are shown in Table 4.4, grouped in pairs between the lines.

Factor-level Combination	Factor Effects								
	I	A	B	AB	C	AC	BC	ABC	Yield
1	1	-1	-1	1	-1	1	1	-1	y_{111}
2	1	-1	-1	1	1	-1	-1	1	y_{112}
3	1	-1	1	-1	-1	1	-1	1	y_{121}
4	1	-1	1	-1	1	-1	1	-1	y_{122}

Table 4.4 The effect representations for the various experimental effects

Columns with letter *I* represent the coefficients for the calculation of the constant effect. The constant effect is simply the overall average response, a measure of location for the response in the absence of any effects due to design factors.

The effects representations in Table 4.4 are grouped in pairs as the effects in each pair are calculated, apart from a change in all the signs, from the same linear combination of the responses. Each effect in a pair is the alias of the other effect in the pair.

For example, the main effect for B and the interaction AB are, respectively,

$$B = \frac{1}{2}(-y_{111} - y_{112} + y_{121} + y_{122}) \qquad \text{Equation 4.2}$$

And

$$AB = \frac{1}{2}(+y_{111} + y_{112} - y_{121} - y_{122}) \qquad \text{Equation 4.3}$$

Hence,

$$B = - AB \qquad \text{Equation 4.4}$$

The negative sign in the equation above indicates that the two effects representations are identical except that one has all its signs opposite those of the other.

Similarly,

I = -A, B = -AB, C = -AC, BC = -ABC

Thus, this fractional factorial experiment results in each experiment effect being aliased with one other experimental effect. No information is available on the main effect of temperature (factor A), because only one of its levels was included in the design. This is why temperature is aliased with the constant effect.

Confounding of effects must be planned so that the effects of most interest are either unconfounded or confounded only with effects that are believed to be negligible relative to the uncontrolled experimental error. When prior knowledge about the magnitude of effects is not available, attention is concentrated on the confounding of main effects and low-order interactions only with high order interactions.

4.5 Analyzing the Results of a $2^{(k-p)}$ Experiment

The ANOVA table has been used to identify the most important factors and interactions on the dependent variables or response variables. The ANOVA table includes:

- Test statistical value (T)
- Test of null hypothesis (P-values)
- Degree of freedom and error component

It is an important tool to determine the significant factors or their interactions on the response variables as well as their level of importance. The factor effect that is not significant is normally distributed with mean zero and variance σ^2.

A variable with a significant effect will have higher ‘T’ value compared to non-significant variables. The sign of the effect determines which factor results in a higher response measurement. A positive sign indicates that the high factor setting results in a higher response

than the low setting; while a negative sign indicates that the low factor setting results in a higher response than the high setting. The level of significance of the variable considered is estimated by calculating the probability of the null hypothesis (P). MINITAB is used to calculate these statistical values.

Adding more factors for the regression model may increase the sum of squares for regression. However, having unimportant factors in the model increases the mean square of error which reduces the usefulness of the model. Therefore, only important interactions are selected as a significant interaction will influence how the main effects are interpreted. Hypothesis testing has to be done to select the most important factors and interactions. A null hypothesis is made as 'the particular factor does not have a significant effect on the response'. The probability of this null hypothesis (P-value) is calculated with respect to the hypothesis made. The calculated P-value is then compared with $\alpha = 0.05$ to validate whether the hypothesis is true or not. If the P-value is less than 0.05 ($P < \alpha$), it means that the null hypothesis is not true. Therefore, the factor or the interaction has a significant effect on the response with more that 95 % significance.

For each term in the model, there is a coefficient. These coefficients are used to construct an equation representing the relationship between the response and the factors.

To use this equation, put in the coded factor values and calculate the predicted response. Because the coefficients are estimated using coded units, putting uncoded factor values into this equation would yield incorrect predictions about strength.

The R^2 value is a commonly-used technique to check the validity of a model. The R^2 value is calculated using SSE and SST. This technique will be used to validate process models. The calculated value for R^2 value may vary from zero to one.

$$R^2 = 1 - \frac{SSE}{SST} \qquad \text{Equation 4.5}$$

R^2 is used to compare the experimental data and model predicted data. If R^2 is close to 100 %, this indicates that the model significantly elaborates the total population of the data and hence validates that the model is acceptable. Adding more variables to the model will increase the R^2 significantly. Therefore, the adjusted R^2 is calculated to check the validity of the model

further. If the adjusted R^2 is lower than R^2, this means that the model does not adequately predict the process. This normally means that some explanatory variables are missing. Without them, the variation in the dependent variable is not fully measured. The equation below shows the adjusted R^2 calculated:

$$R^2_{adj} = 1 - \frac{n-1}{n-p}(1 - R^2) \qquad \text{Equation 4.6}$$

Where,

n is the number of observations made

p is the number of model parameters

4.6 Graph Options

4.6.1 Effect Plot

A normal effects plot is used to compare the relative magnitude and the statistical significance of both main and interaction effects. MINITAB draws a line to indicate where the points would be expected to fall if all effects were zero. Points that do not fall near the line usually signal significant effects. Such effects are larger and generally further from the fitted line than unimportant effects. By default, MINITAB uses a α-level of 0.05 and labels any effect that is significant.

Figure 4.2 shows an example of the effect plot:

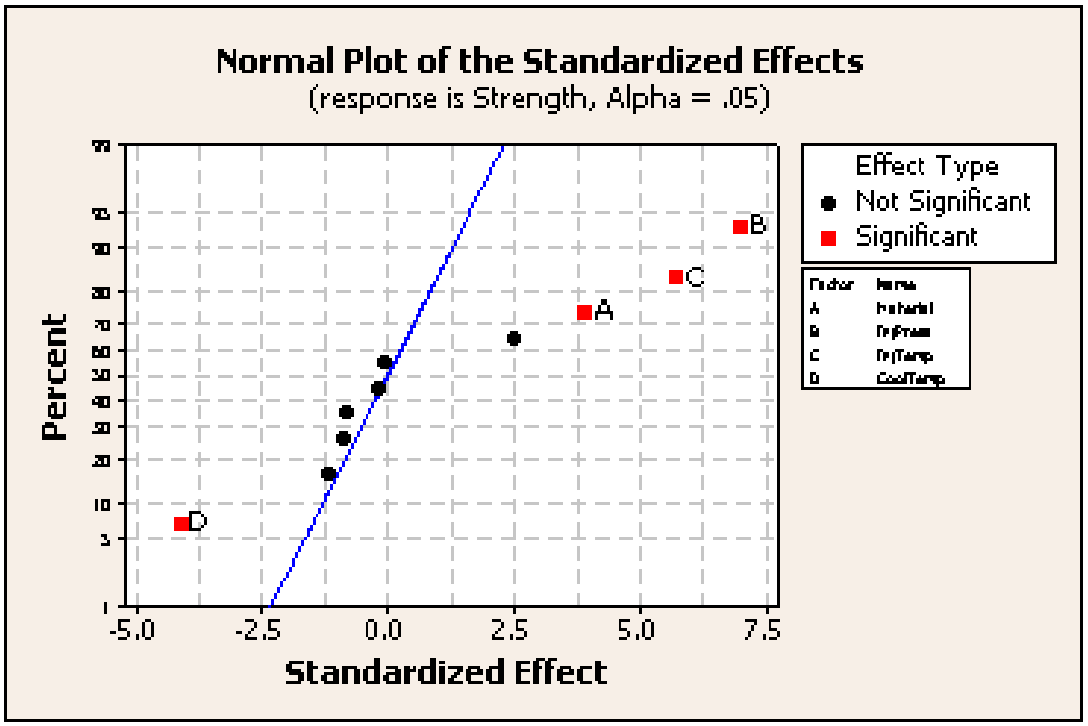


Figure 4.2 Example of Normal Plot

The plot indicates the direction of the effect. Factors A, B and C all have positive effects because they reside to the right of the line. This means that when changes from the low level to the high level of the factor are made, the response increases. However, as factor D resides to the left of the line, it has a negative effect, meaning that when changes are made from the low level to the high level of the factor, the response decreases.

4.6.2 Pareto chart of effects

A Pareto chart of the effects is normally used to compare the relative magnitude and the statistical significance of both main and interaction effects. MINITAB displays the:

- absolute value of the unstandardized effects when there is not an error term
- absolute value of the standardized effects when there is an error term

MINITAB plots the effects in decreasing order of the absolute value of the effects. The reference line on the chart indicates which effects are significant. When the model contains

an error term, by default, MINITAB uses a α-level of 0.05 to draw the reference line. With no error term, MINITAB uses Lenth's method to draw the reference line.

Figure 4.3 demonstrates an example of a Pareto chart of effect:

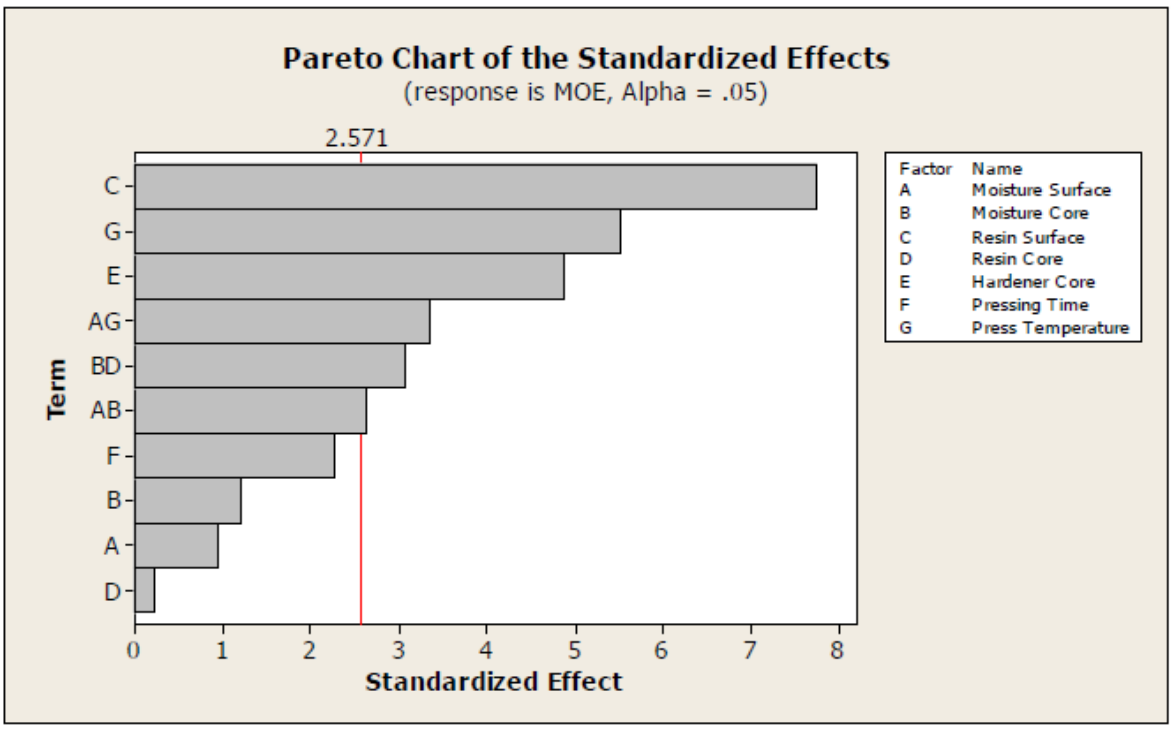


Figure 4.3 Example of Pareto Chart

4.6.3 Surface and contour plots

Contour plots can be used to visualize the response surface. Contour plots are useful for establishing desirable response values and operating conditions.

This plot shows how a response variable relates to two factors based on a model equation. Points that have the same response are connected to produce contour lines of constant responses. Since a contour plot shows only two factors at a time, while holding any other factors and covariates at a constant level, contour plots are only valid for fixed levels of the extra factors. If the holding levels are changed, the response surface changes as well, sometimes drastically.

Figure 4.4 shows an example of a contour plot:

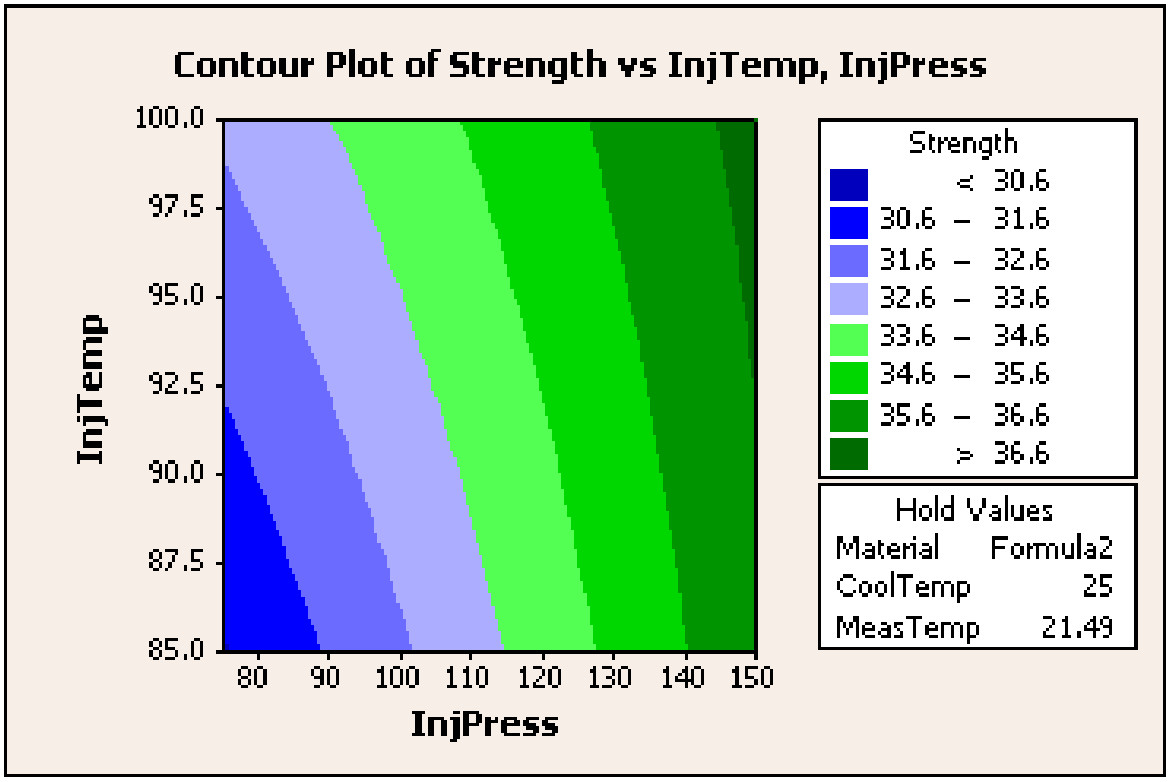


Figure 4.4 Example of Contour Plot

This plot shows how the injection pressure and injection temperature are related to insulation strength. This plot can only be used for material with Formula 2, cool temperature at 25 and the covariate measured temperature at its mean 21.49. The darkest green area indicates the region where the response is higher. In order to optimize the insulation strength, it is

recommended to use the settings for injection pressure and injection temperature in the upper right corner of the plot (Example from MINITAB 15).

4.7 Application of Design of Experiments (DOE) in Various Industries

Design of experiment (DOE) is widely used in research as well as in industrial settings, although sometimes for very different purposes. The primary goal in scientific research is usually to show the statistical significance of an effect that a particular factor exerts on the dependent variable of interest. In industrial settings, the primary goal is usually to extract the maximum amount of unbiased information regarding the factors affecting a production process from as few observations as possible.

Experimental design is increasingly used in manufacturing to optimize the production process. Specifically, the goal of this method is to identify the optimum settings for the different factors that affect the production process. Many researchers in the past used this method for their investigations. The following section provides some examples from industry.

Design of Experiments Helps Optimise Injection Moulding of Conductive Compounds (Plastics Today, 2011)

RTP Company, a leading compounder of custom-engineered thermoplastics, recently used design of experiments (DOE) to determine which injection moulding process conditions optimize conductive properties for a particular material. DOE made it possible to explore the complete processing space and provide users with a formula to calculate the conditions that would deliver the required resistivity levels. The predictive capabilities of DOE made it possible to fully explore the processing window in fewer runs.

In the above study, DOE helped to set the processing conditions to achieve the required levels of surface resistivity with the materials. It also showed which process conditions required accuracy and which ones could have variability. It also provided significant statistical information that helped to demonstrate its validity.

Production of Particleboard from Wheat Straw (Luo and Yang, 2010)

DOE was used to investigate the suitability of wheat straw for particleboard production. Poly-isocyanate resin was used as a binder. The variables studied and their respective variation ranges were: hot-press temperature, 180~200°C; hot-press time, 3 - 7 min; resin application ratio, 4 % to 6 %, wax application ratio, 0. 4% to 1.0 %. After analysis of DOE, the optimum manufacture conditions were found to be 190°C for hot-press temperature, 5 min for hot-press time, 6 % for resin application ratio, 0.7 % for wax application ratio. Under the optimum conditions, 12.3 MPa of the modulus of rupture, 0.33 MPa of the internal bond and 4.5 % of the thickness swell were obtained, higher than the requirement of JIS A 5908.

Hot-pressing Process Optimization by Response Surface Methodology (Park et al. 1999)

Experimental design combined with regression analysis to develop a response model has been used on many occasions to find the unknown mechanisms between response and the process. In this study, regression modelling was used to model the hot pressing process of the three-layer MDF production process. First, they found the most important factors with respect to internal temperature of the MDF during production. Then these factors were used to find the relationships for temperature profile and density profile with respect to pressing time, press temperature and moisture content. The developed models were then used to optimise the hot pressing process of the fibre board.

4.8 Summary

This chapter has introduced the DOE method and explained its efficiency in developing new products using new materials, or improving existing products. 2^k fractional factorial design is recommended to design experiments and analysis. ANOVA is utilized to identify the most important factors and their interaction for developing a new product. This chapter has also demonstrated that DOE methods have been used by various researchers in the past to develop new materials, to develop experiments with new materials and to optimize existing processes.

In the work presented in this thesis, DOE has been adopted in its various forms to optimize particleboard properties of boards made of mixtures of hardwood and softwood residues.

Chapter 5

GENERAL PROCEDURE FOR PRODUCING PARTICLEBOARD IN THE LABORATORY AND METHODS OF TESTING

5.1 Overview

After finalising the experimental design, some preliminary studies on the particleboard production procedure in the laboratory were needed prior to the planning of screening tests. This process was essential to develop the experimental procedures in detail.

5.2 Introduction

This chapter presents the procedures, materials and equipment used for the particleboard production research in the RMIT laboratories. The calculations of various mix proportions derived from experimental designs with a number of variables are also presented. Further, this chapter discusses the testing methods adopted to investigate the physical and mechanical properties of the board produced.

5.3 Materials Used

5.3.1 Hardwood Residues

As explained in Chapter 4, experiments were conducted with different mix proportions incorporating hardwood and softwood residues. The hardwood residues were supplied by Dormit Timber Pty Ltd of Dandenong South, Victoria. The hardwood timbers processed by Dormit Timber Pty Ltd were a species of Eucalyptus. They are relatively high in density, ranging from 680 to 870 kg/m^3 (Bootle, 1983). The shape of hardwood residues is most likely rectangular and cubic. Also, they are acidic, and the pH value ranges from 3.5 to 4.5.

Sawmill residues come in two types: mulch and fine. Mulch, as shown in Figure 5.1, has bigger particles including both cubical and flake-shaped particles. Figure 5.2 shows fine residue with smaller particles mainly in cubic shapes. 95 % of the mulch contains particles bigger than 5 mm, whilst 90 % of the fine residue contains particles smaller than 5 mm.

Figure 5.1 Hardwood Mulch

Figure 5.2 Hardwood Fine

Mixing 45 % Mulch with 55% Fine would provide a relatively uniform particle size distribution, which is suitable for the core layer. For the surface layer, mixing 35 % Mulch with 65 % Fine that contains higher amounts of smaller particles is suitable, as the softwood particleboard industry generally uses smaller particles for the surface layer and bigger particles for the core layer.

5.3.2 Softwood Residues

The softwood residues were supplied by D & R Henderson Pty Ltd of Benalla, Victoria. These particles are commonly used by D & R Henderson for particleboard production. Softwood residues are relatively smaller than the hardwood residues, as shown in Figure 5.3 and Figure 5.4.

The mulch and fine residues were oven dried at 105^{o}C for at least 48 hours to remove the moisture. Then, these particles were measured and mixed separately in accordance with the mix proportions identified for the surface and for the core.

Figure 5.3 Softwood Mulch

Figure 5.4 Softwood Fine

5.3.3 Resin and Hardener

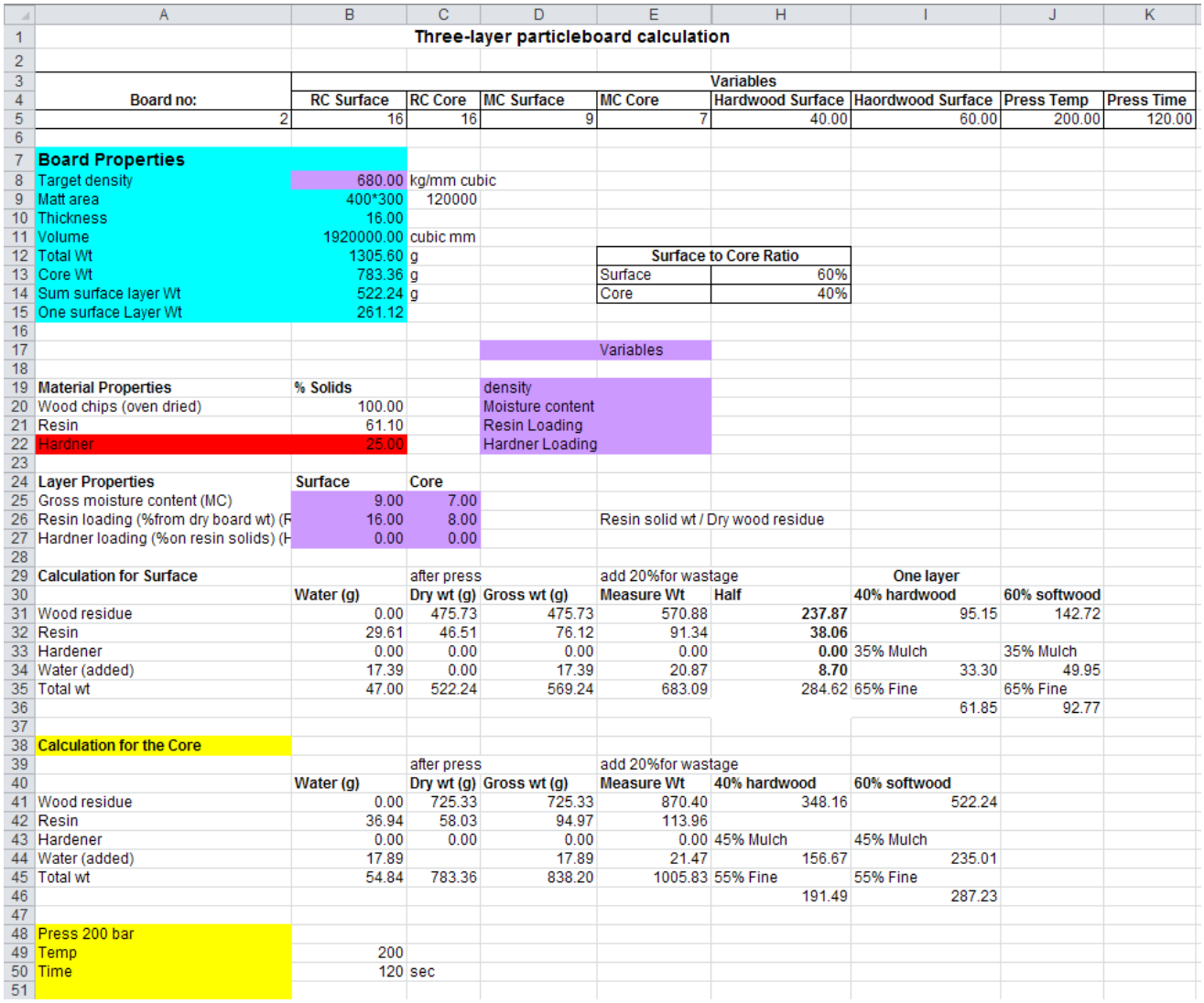
Urea Formaldehyde (UF) was selected as a bonding agent for the particleboard production in the RMIT laboratory. UF is one of the most commonly used resins in softwood particleboard manufacturing due to its lower market value, durability and performance. The chemical division of Hexion Pty Ltd provided the UF resin in liquid form with 63 % to 65% solids, and viscosity in the range of 115 – 220 cPs at 25^{o}C.

Hardener is normally used in the softwood particleboard industry to create an acidic medium to accelerate the curing of UF resin and shorten the pressing time. Hardener is generally used for the core layer at 1 % to 2 % only. However, as mentioned earlier, the hardwood residue used in the research was already acidic. Therefore, hardener was not required for the purposes of the research.

5.4 Particleboard Mix Proportion

Since there are several variables which affect the properties of particleboard, each single board produced from the laboratory had different combinations of variables, in accordance with the screening test table generated from MINITAB. The calculation of mix proportions was essential to ensure the appropriate proportions of wood particles, resin and water were used during the production.

Figure 5.5 presents the spreadsheet developed for the calculation of particleboard mix proportions.



Three-layer particleboard calculation

Board no:	Variables: RC Surface	RC Core	MC Surface	MC Core	Hardwood Surface	Haordwood Surface	Press Temp	Press Time
2	16	16	9	7	40.00	60.00	200.00	120.00

Board Properties

Target density	680.00	kg/mm cubic
Matt area	400*300	120000
Thickness	16.00	
Volume	1920000.00	cubic mm
Total Wt	1305.60	g
Core Wt	783.36	g
Sum surface layer Wt	522.24	g
One surface Layer Wt	261.12	

Surface to Core Ratio	
Surface	60%
Core	40%

Variables

Material Properties	% Solids
Wood chips (oven dried)	100.00
Resin	61.10
Hardner	25.00

density
Moisture content
Resin Loading
Hardner Loading

Layer Properties	Surface	Core
Gross moisture content (MC)	9.00	7.00
Resin loading (%from dry board wt) (F	16.00	8.00
Hardner loading (%on resin solids) (H	0.00	0.00

Resin solid wt / Dry wood residue

Calculation for Surface

	Water (g)	after press: Dry wt (g)	Gross wt (g)	add 20%for wastage: Measure Wt	Half	One layer: 40% hardwood	60% softwood
Wood residue	0.00	475.73	475.73	570.88	**237.87**	95.15	142.72
Resin	29.61	46.51	76.12	91.34	**38.06**		
Hardener	0.00	0.00	0.00	0.00	**0.00**	35% Mulch	35% Mulch
Water (added)	17.39	0.00	17.39	20.87	**8.70**	33.30	49.95
Total wt	47.00	522.24	569.24	683.09	284.62	65% Fine	65% Fine
						61.85	92.77

Calculation for the Core

	Water (g)	after press: Dry wt (g)	Gross wt (g)	add 20%for wastage: Measure Wt	40% hardwood	60% softwood
Wood residue	0.00	725.33	725.33	870.40	348.16	522.24
Resin	36.94	58.03	94.97	113.96		
Hardener	0.00	0.00	0.00	0.00	45% Mulch	45% Mulch
Water (added)	17.89		17.89	21.47	156.67	235.01
Total wt	54.84	783.36	838.20	1005.83	55% Fine	55% Fine
					191.49	287.23

Press 200 bar	
Temp	200
Time	120 sec

Figure 5.5 Spreadsheet for calculation of particleboard recipes

The table in blue at the top of Figure 5.5 represents the board number with the values of variables extracted from the screening test table. These variables are resin content in surface

layer, resin content in core layer, moisture content in surface layer, moisture content in core layer, hardwood portion in surface layer, hardwood portion in core layer, pressing temperature and pressing time.

The target board properties are clearly stated in Row 7 to Row 15. Target board density was expected to be 680 kg/m^3 as reference to the normal board density (Table 3.1). The total matt area was 400 x 300 mm according to the dimension of the mould. A stopper of 16 mm was used during the press to ensure that the board thickness remained at 16 mm. With the thickness and area of the board, volume of board can be calculated as:

$$V = A \times t \qquad \text{Equation 5.1}$$

Where,

V = volume of board, mm^3

A = area of board, mm^2

t = thickness of board, mm

The total weight of the board can be expressed as follows:

$$W_{total} = \rho_{target} \times V \times 10^{-6} \qquad \text{Equation 5.2}$$

Where,

W_{total} = total weight of board, g

ρ_{target} = target board density, kg/m^3

V = volume of board, mm^3

Based on previous research (Nirdosha, 2007), surface layers of particleboard contribute 40 % (refer to cell H14) of the total weight; while the core layer contains 60 % (refer to cell H13) of the total weight. Therefore, the weight of core and surface layers can be simply calculated as:

$$W_{core} = 60\% \times W_{total} \qquad \text{Equation 5.3}$$

The total weight is also the combination of the weights of the solid resin, hardener and dry wood residue. Since the particles were dried in the oven before measurement, they consist of 100 % solid weight. The resin and hardener consisted of 61.1 % and 25 % of solid weight, respectively (refer to cell B20 to B22). This indicates that resin and hardener consist of 38.9 % and 75 % of water weight, respectively.

For the surface layer properties, water, resin and hardener are added during the process of making particleboard. In the spreadsheet for board no. 2, the amount of water added is 9 % (refer to cell B25) of the total weight. The amount of resin required is 16 % (refer to cell B26), from the weight of dry board (see cell D31). Hardener is ignored as it is not required in this process, but whenever it is needed, the amount of hardener is measured based on the solid weight of resin.

The calculation of the weight of the dry wood residue, $W_{p(solid)}$ (cell D31) is calculated first. Since the total weight is the combination of the weights of the solid resin, hardener and dry wood residue, the equation for total weight can also be expressed as:

$$W_{total} = W_{r(solid)} + W_{h(solid)} + W_{p(solid)} \qquad \text{Equation 5.4}$$

Where,

$W_{r(solid)}$ = the solid weight of resin (cell D32), g

$W_{h(solid)}$ = the solid weight of hardener (cell D33), g

$W_{p(solid)}$ = the solid weight of wood residues, g

Taking into consideration the solid percentage of each material, Equation 5.4 can be presented as:

$$W_{total} = 0.611W_r + 0.25W_h + W_p \qquad \text{Equation 5.5}$$

Where,

W_r = measured weight of resin, g

W_h = measured weight of hardener, g

W_p = measured weight of wood residues, g

Dividing Equation 5.5 by W_P, we obtain:

$$\frac{Wtotal}{Wp} = 0.611\frac{Wr}{Wp} + 0.25\frac{Wh}{Wp} + 1 \qquad \text{Equation 5.6}$$

To determine the solid weight of wood residue, Equation 5.6 will be written as:

$$W_p = \frac{Wtotal}{(0.611\frac{Wr}{Wp}+0.25\frac{Wh}{Wp}+1)} \qquad \text{Equation 5.7}$$

W_r/W_p is the solid weight ratio of resin and wood residues. From Figure 5.1, the value of W_r/W_p can be calculated as:

$$\frac{Wr}{Wp} = \frac{16\%}{100\%} = 0.16 \qquad \text{Equation 5.8}$$

W_h/W_p is the solid weight ratio of hardener and wood residues, but the solid weight of hardener is based on the solid weight of resin. Therefore, W_r/W_p should be inserted into Equation 5.7 and the resulting equation expressed as:

$$W_p = \frac{Wtotal}{(0.611\frac{Wr}{Wp}+0.25\frac{Wh}{Wr}x\frac{Wr}{Wp}+1)} \qquad \text{Equation 5.9}$$

In figure 5.1, the value of W_h/Wr is

$$\frac{0.25Wh}{0.611Wr} = \frac{0\%}{100\%}$$

Therefore,

$$\frac{Wh}{Wr} = \frac{0\%}{100\%}\chi\frac{0.611}{0.25} = 0$$

Hence, the weight of hardener, W_h is zero. The weight of wood residues, W_p can be easily presented as:

$$W_p = \frac{Wtotal}{(0.611\frac{Wr}{Wp}+1)} \qquad \text{Equation 5.10}$$

After obtaining the weight of wood residue, the weight of resin, W_r (cell D32) can simply be calculated from Equation 5.8, as below:

$$W_r = 0.16 \times W_p \qquad \text{Equation 5.11}$$

From Equations 5.4 and 5.5, the solid weight of resin, $W_{r(solid)}$ (cell C32) can be determined as:

$$W_{r(solid)} = 0.611W_r \qquad \text{Equation 5.12}$$

The solid weight of hardener, $W_{h(solid)}$ can be calculated as:

$$W_{h(solid)} = 0.25W_h \qquad \text{Equation 5.13}$$

The wood residues were fully oven-dried before measured and hence there was no moisture content in them, as indicated in cell B31. As mentioned earlier, the resin consisted of 38.9 % of water weight. Therefore, the water weight of resin, $W_{r(water)}$ (cell B32) can be represented as:

$$W_{r(water)} = 0.389W_r \qquad \text{Equation 5.14}$$

For board no. 2, the total weight of water of the materials, $W_{total(water)}$ (cell B35), was set to be 9 % of the weight of the surface layer. It can be determined as:

$$W_{total(water)} = 0.09W_{total} \qquad \text{Equation 5.15}$$

Also,

$$W_{total(water)} = W_{r(water)} + W_{h(water)} + W_{w(added)} \qquad \text{Equation 5.16}$$

Where,

$W_{w(added)}$ = the amount of water added (cell B34), g

$W_{h(water)}$ = the water weight of hardener (cell B33), g

Therefore, the amount of water added is

$$W_{w(added)} = W_{total(water)} - W_{h(water)} + W_{h(water)} \qquad \text{Equation 5.17}$$

The gross weight of solid and water weight of the materials is shown in cell D35. Due to the wastage during the process of making particleboard, 20 % of wastage is added to the gross weight, which is indicated as measured weight in cell E30 to E35.

For board no. 2, 40 % of hardwood residue and 60 % of softwood were selected for the surface layer. Therefore, 60 % of total wood residue weight in the surface layer will be hardwood residues, as calculated in cell I31:

$$W_{hardwood} = 0.4 \times W_{p(measured)} \quad \text{Equation 5.18}$$

40 % of total wood residue weight in the surface layer will be softwood as in cell J31:

$$W_{softwood} = 0.6 \times W_{p(measured)} \quad \text{Equation 5.19}$$

Within the surface layer, 65 % of particles are fine while 35 % of particles are mulch. Therefore, the amounts of mulch and fine in $W_{hardwood}$ and $W_{softwood}$ can be calculated respectively as:

$$W_{fine} = 0.65 \times W_{hardwood} \quad \text{Equation 5.20}$$

And

$$W_{mulch} = 0.35 \times W_{hardwood} \quad \text{Equation 5.21}$$

Where,

W_{fine} =the weight of fine particles (cell I36), g

W_{mulch} = the weight of mulch particles (cell I34), g

A similar calculation method can be applied for the core layer to obtain appropriate measurements of materials for particleboard production in the laboratory.

5.5 Apparatus and Procedures used to Manufacture Particleboard in Laboratory

This section outlines the major equipment used to produce particleboard in the School of Civil and Chemical Engineering laboratory at RMIT University, Melbourne.

5.5.1 Manufacturing Apparatus

Mixer Drum and Spray Gun

A concrete mixer with a capacity of 2.2 cubic feet was used to mix the particles with resin as shown in Figure 5.6. The rotating speed of the mixer is 1250 rpm. The lid is designed with a hole of a diameter of 8 cm which allows resin to be sprayed into the mixer.

Figure 5.6 Mixer Drum

A spray gun with a high pressure pot was used to spray the resin into the mixer while mixing particles. It is a high pressure spray gun with a nozzle diameter of 1.5 mm as shown in Figure

5.7. The pot capacity is 1 litre, and it has a pressure capacity up to 100 kPa. A small container with the mix of resin and water was kept inside the pressure pot to reduce wastage as well as assist cleaning the container quickly in order to switch efficiently from one mix to another. A constant pressure of 80 kPa was maintained during all mixing.

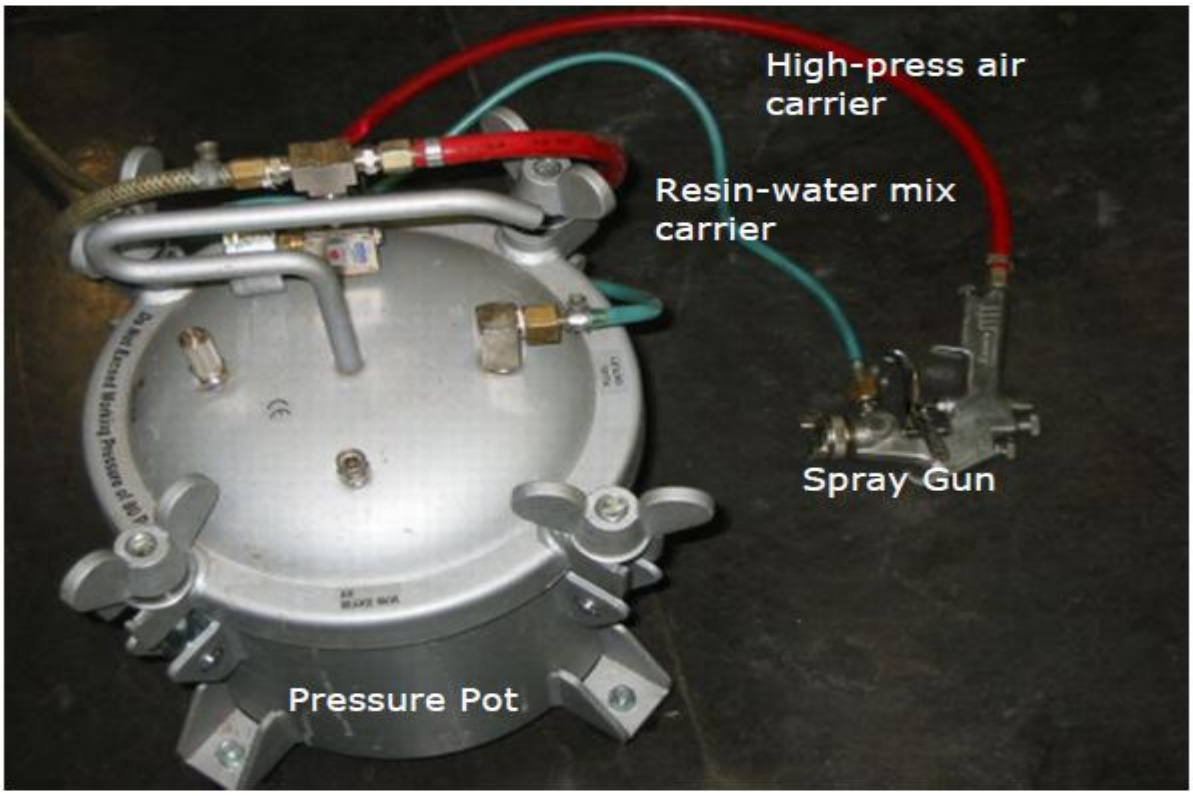


Figure 5.7 Pressure Pot and Resin Spray Gun

Mould

A casting mould with a mould area of 300 x 400 mm was used for particleboard casting before pressing. The depth of the casting mould was 120 mm. The casting mould is shown in Figure 5.8. The lid was used to level the particles of each layer. Two 3 mm thick aluminium plates were used as a protection against the particle pulp. The area of each aluminium plate was 500 x 500 mm. They were placed on the top and bottom of the particle pulp in order to ensure consistent heat transmission from both top and bottom. Teflon foil was placed on both top and bottom aluminium plates during the moulding to stop adherence of the top and bottom of the final board to the aluminium plates after hot pressing.

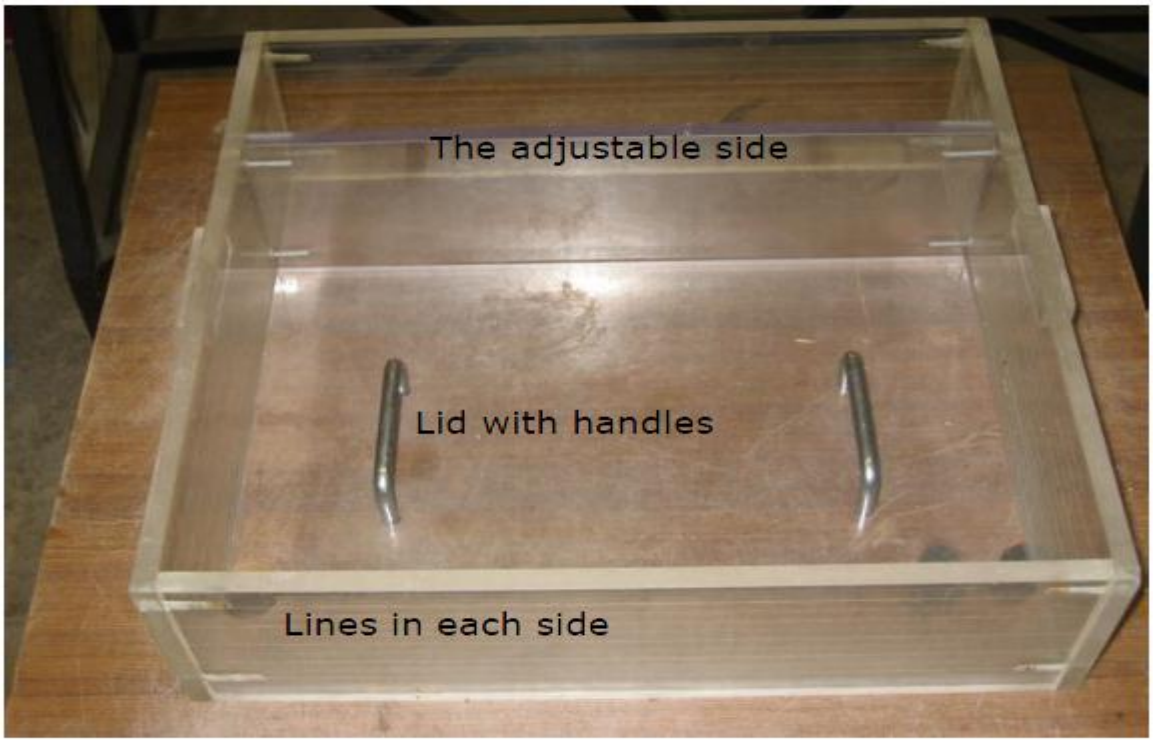


Figure 5.8 Casting Mould

Cold Press and Hot Press

A 'Wabash' hot press machine, from Vickers Ruwolt Pty Ltd was used as shown in Figure 5.9. It contains two mounted platens for pressing particlepulp. The pressure capacity of the hot press machine was 40 tons on 5" of ram diameter, with a heating temperature up to 400^{o}C. It takes approximately an hour to reach 190^{o}C. This machine has no thickness controller, but it has a temperature controller. Therefore, the final desired thickness of particleboard has to be achieved by using a steel rod of 16 mm thickness as a thickness stopper. The effective area for the hot press machine is 500 x 500 mm.

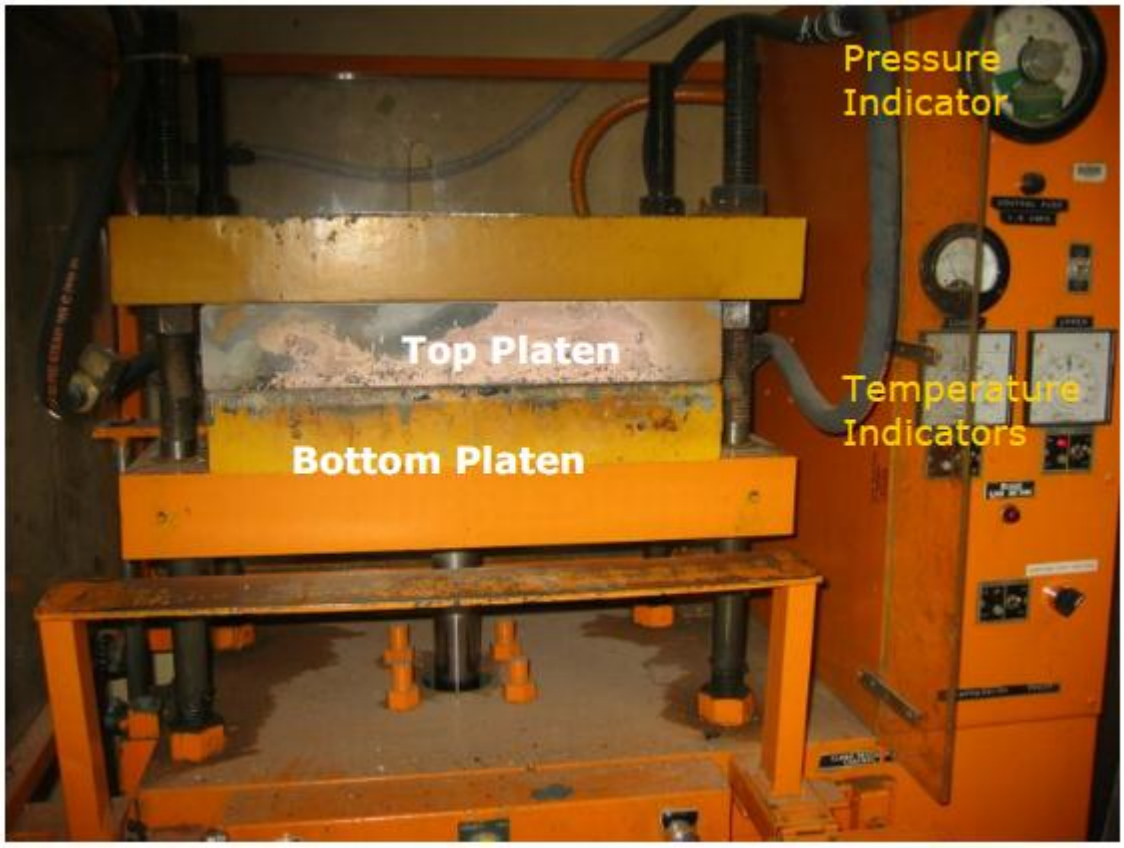


Figure 5.9 'Wabash' Hot Press

Figure 5.10 shows the cold press machine that was used. It is a high pressure hydraulic press with a pressing capacity of 25 tons on 5" ram diameter. The maximum press area for the press is 500 x 500 mm. A stopper at 20 mm thickness was used to control the thickness of particle pulp during cold pressing.

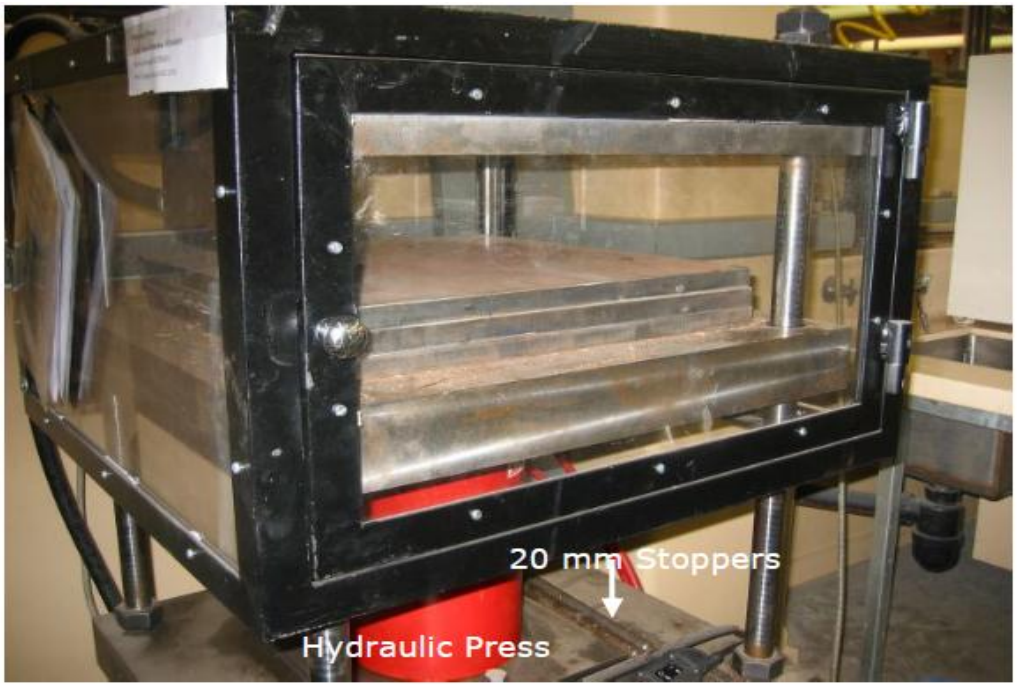


Figure 5.10 Hydraulic Cold Press

Humidifier

A humidifier was used for specimen conditioning before testing was carried out. Figure 5.11 shows the humidifier used for the project. It is able to control relative humidity and temperature in the range of +10 ^{o}C to +60 ^{o}C and 30 % to 90 % respectively.

Figure 5.11 Humidifier

5.5.2 Manufacturing Procedure

In this section, the procedure for making particleboard in the laboratory will be explained. The process is derived from the softwood particleboard production industry. There are four main steps in making particleboard: sampling and conditioning, mixing the materials, pre-pressing and hot pressing of specimens, curing and storage of specimens.

Sampling and Conditioning

The wood residues collected from the supplier contained very high initial moisture content at around 80 %. For this reason, the residues were oven dried at a temperature of 105^{o}C for at least 24 hours, as shown in Figure 5.12. The particles were then allowed to cool for one hour in the normal environment before the mixing of particles was carried out.

Figure 5.12 Oven-drying wood residues

Mixing

The amounts of mulch and fine residues for both surface and core layers were first measured respectively in accordance with the calculated amounts. The mulch: fine ratio for the surface layer was maintained at 35 %: 65 % and for the core at 45 %: 55 % (by weight). After fine and mulch were weighed, they were mixed in the mixing drum for 2 minutes prior to spraying the resin mix.

While mixing the particles, the calculated amounts of resin and water required for each layer were measured then mixed in a small container. The mix was then placed into the pressure pot for spraying. The mix was then sprayed into the mixing drum as shown in Figure 5.13. The mixing drum continued to operate for 5 minutes after completion of spraying to ensure complete mixing of resin and particles. For occupational health and safety reasons, an exhaust system was in operation at all times while particles were being resinated. The pot was washed after each spraying to prevent clogging.

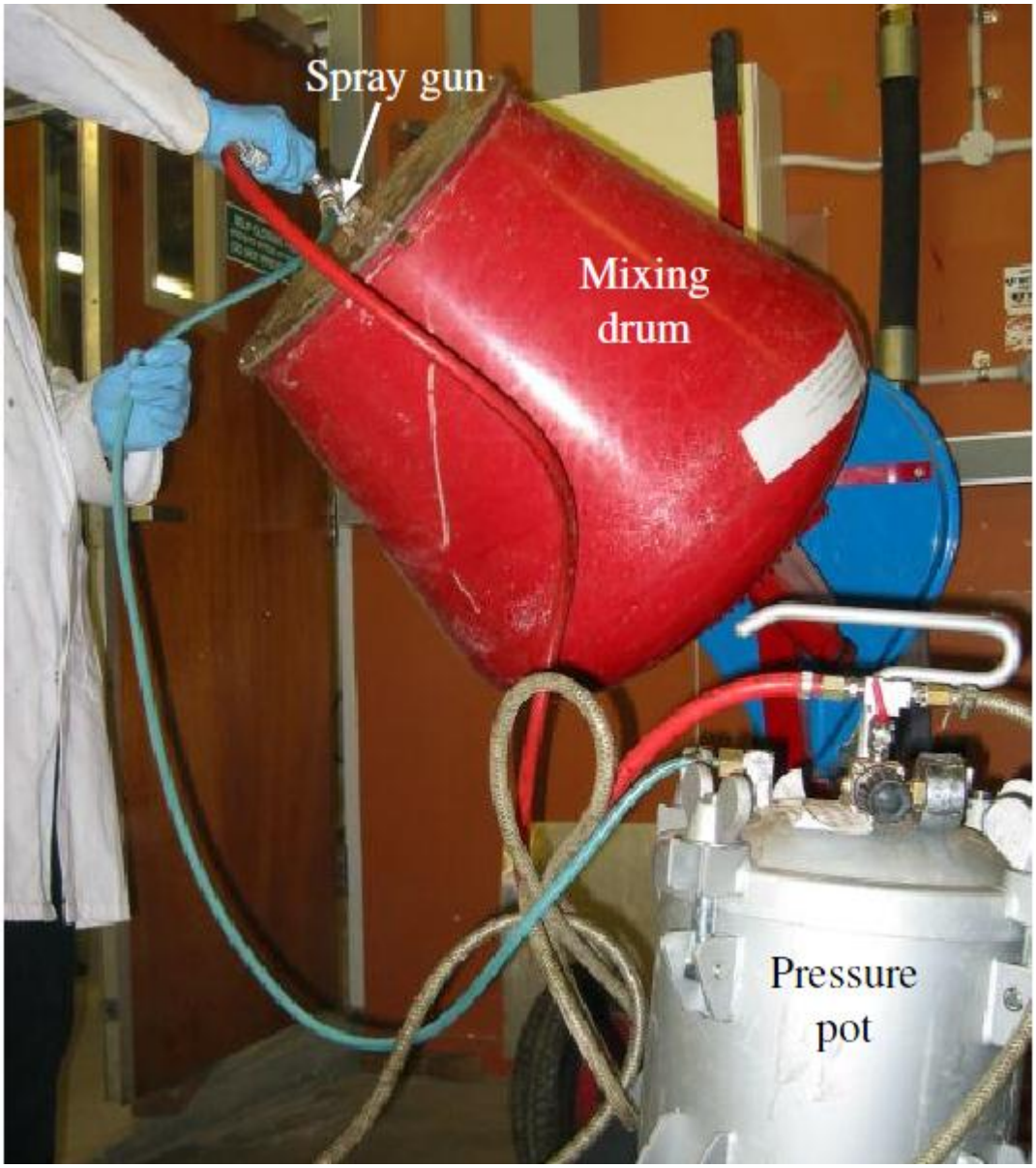


Figure 5.13 Mixing procedure

Moulding and Pressing

After mixing, the pulp was ready for moulding before pressing. The pulp for the surface layer was weighed and separated into containers for the two surface layers; while the pulp for the core layer was placed in another container. Moulding then commenced in accordance with the following procedures:

- An aluminium plate was placed on the bench.
- Teflon foil was then spread on the aluminium plate.

- One surface layer material was spread first, followed by the core layer material and ending with the top surface layer.
- The thickness and level of each layer were maintained with the help of lines in the walls of the mould.

Once the moulding was finished, the mould was compressed by standing on the lid, as shown Figure 5.14. Before the mat was transferred to cold press, the lid was taken away from the pre-pressed mat. Another Teflon foil was then placed on top of the mat, followed by an aluminium plate.

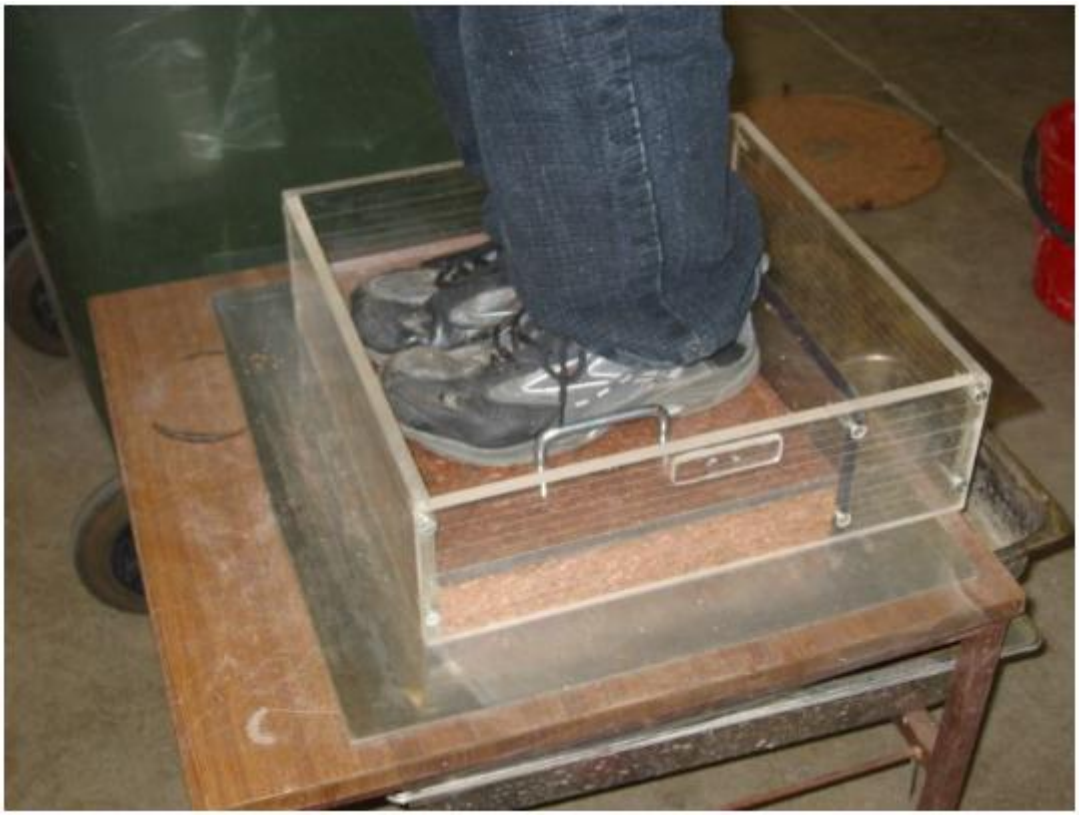


Figure 5.14 Pre-press mat

The moulded mat was then transferred into the cold press machine as shown in Figure 5.15. Constant pressure was maintained on each board during the cold pressing operation. Cold pressing is important to remove trapped air from the wood mat. Two steel stoppers at 20 mm thickness were used to maintain the thickness of the mat. Cold and hot pressing times were kept equal (depending on the experimental design values) so that no machines were idle in order to maintain a low production cost. . After the cold press, the mat was then moved into

the hot press at a selected temperature and for a specified pressing time (Figure 5.16). Stoppers at 16 mm thickness were used to maintain the particleboard at the standard thickness.

Figure 5.15 Cold Pressing

Figure 5.16 Hot Pressing

Curing and Storage of specimens

After hot press, the board was removed and left to cool as shown in Figure 5.17. Spacers were used between boards to facilitate ventilation. The cooled board was then stored in the ventilation area for a week to remove the trapped air and formaldehyde before cutting and sampling.

Figure 5.17 Particleboard for cooling

The particleboards were then cut into the desired sizes before placement in a humidifier for curing and storage (Figure 5.18). The initial dimensions of the particleboard were 300 x 400 mm. In cutting of the particleboards, 50 mm of each edge of particleboard was trimmed off. Two 100 x 300 mm specimens were cut for three-point flexural bending tests, and two 50 x 50 mm specimens were cut for internal bonding tests. After cutting, the samples were placed in the humidifier for at least 24 hours at 20^{o}C with relative humidity of 65 % for conditioning (Figure 5.19).

Figure 5.18 Chainsaw for board cutting

Figure 5.19 Samples conditioning before testing

5.5.3 Testing Apparatus and Test Methods

Internal Bonding (IB) Test

Internal Bonding (IB) testing was carried out in according with AS/NZS 4266.6. The test determines the resistance of tension perpendicular to the surface of the test piece by submitting the latter to a uniformly-distributed tensile force until rupture occurs. The tensile strength perpendicular to the plane of the sample is determined by the maximum load in relation to the surface area of the test piece.

Grips (see Figure 5.20) capable of transmitting the tensile force applied to the test block were prepared. The grips were designed to be self-aligning so that each test block could be positioned perfectly.

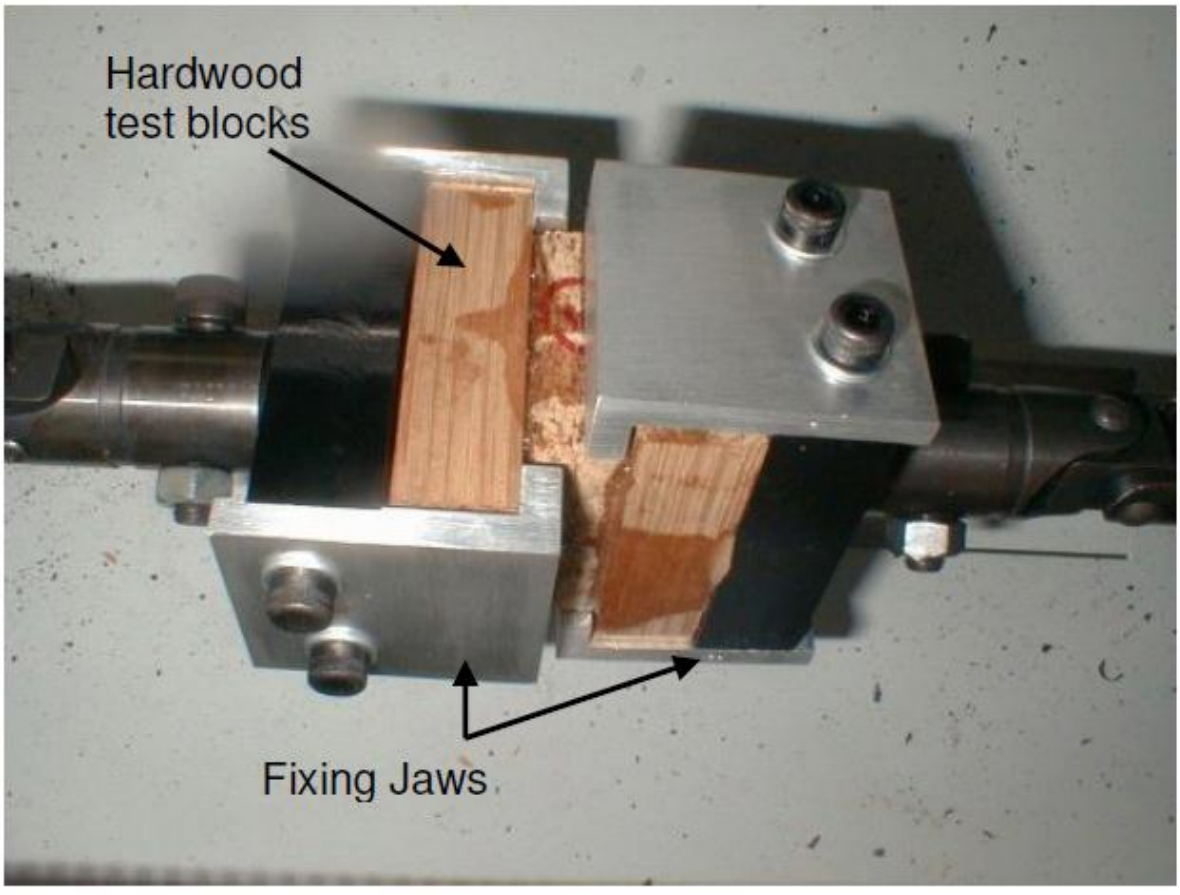


Figure 5.20 IB Testing

Five pieces of 50 x 50 mm test samples were prepared and conditioned. Two loading blocks were prepared for each test sample. Each test piece was then bonded to the loading block

using Araldite epoxy resin. Araldite bonded samples were then conditioned for 24 hours in order to fully cure the Araldite resin. The upper and lower blocks were oriented at 90^{o} as shown in Figure 5.20. The test was not carried out until the Araldite had had sufficient time to cure. Every test piece attached to loading blocks was stored in conditions of 65 % relative humidity and a temperature of 20 ^{o}C for 24 hours for the Araldite to cure.

After curing, each test block was positioned in the grips. The load was then applied at a constant rate until rupture occurred. The rate of loading was adjusted so that the rupture occurred within 60 sec. Once rupture occurred, the maximum load sustained by the test piece was recorded.

Tensile strength perpendicular to the plane of the panel of each test piece (f), was expressed in megapascals to two decimal points, according to the following equation:

$$f = \frac{Fmax}{ab} \qquad \text{Equation 5.22}$$

where,

Fmax = breaking load, in newtons

a, b = length and width of the test piece, in millimetres

These calculated IB values were then recorded for analysis.

Modulus of Elasticity (MOE) Testing

Modulus of elasticity (MOE) was tested in accordance with AS/NZS 4266.5. MOE was determined by applying load to the centre of a test piece supported at two points.

A three-point bending test was carried out to determine MOE, as shown in Figure 5.21. Two parallel metal rollers with a diameter of 25 mm were prepared as supports for the test piece to sit on. A cylindrical loading head was placed parallel to the supports and equidistant from them.

Each the test piece is required to be rectangular, with a width of not less than 50 mm and a length at least 15 times the nominal thickness plus 50mm. Therefore, a sample size of 100 x 300 mm was selected for the test. The test pieces were conditioned in accordance with AS/NZS 4266.1.

Testing was then carried out by placing the test piece horizontally on parallel metal rollers with a diameter of 25 mm which were free to rotate. The centre spacing of the rollers was maintained at 250 mm. Then, a load normal to the face of test piece was applied to the centre of the test piece at a constant rate. The deflection at a given load was measured accordingly. Once sample failure occurred, maximum load was recorded for further calculation.

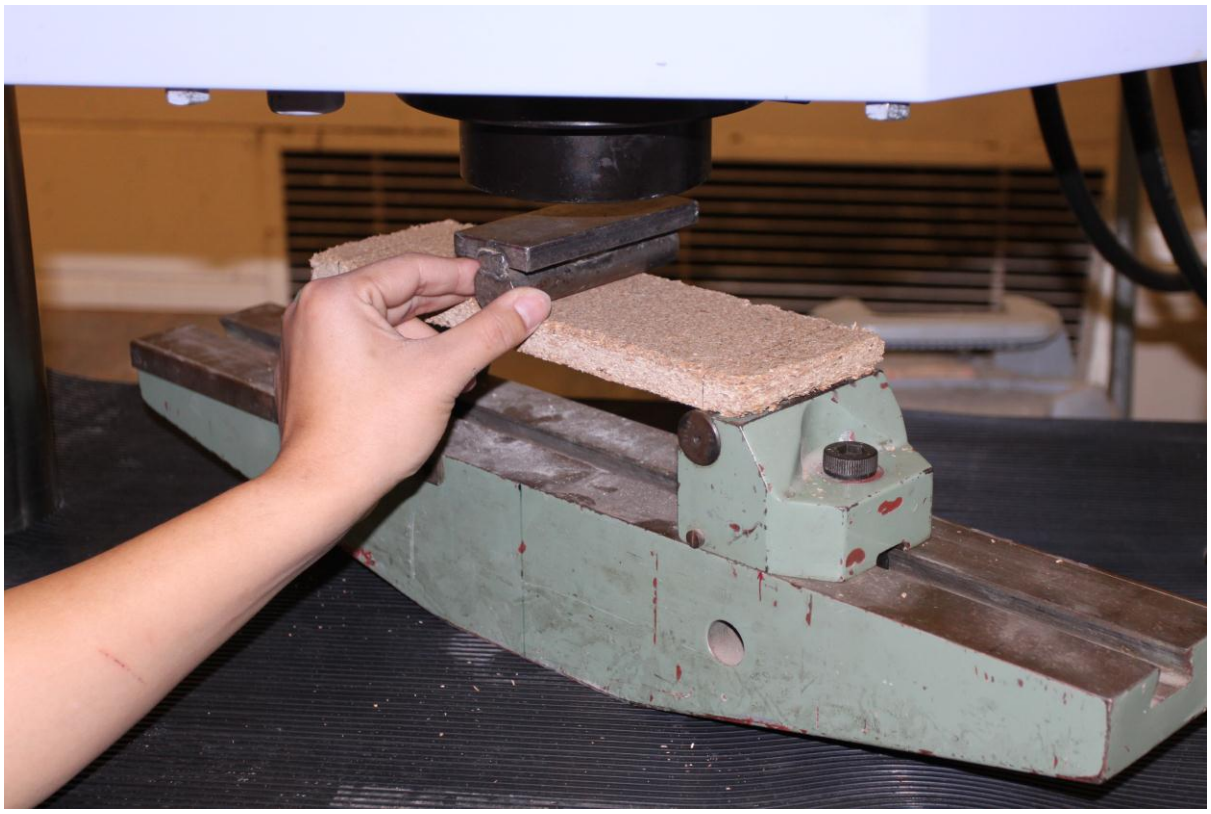


Figure 5.21 Testing of MOE and MOR

The MOE of each sample was calculated using Equation 5.23 considering the corresponding deflection within a load range up to one third of breaking load of the board under test. Therefore, ΔW was considered to be calculated as 40 % of fracture load – 10 % of fracture load.

$$MOE = \frac{L^3}{4bt^3} \times \frac{\Delta W}{\Delta S} \qquad \text{Equation 5.23}$$

Where,

MOE = Modulus of elasticity, in megapascals

L = Distance between the centres of the supports, in millimetres

b = width of the test piece, in millimetres

t = thickness of the test piece, in millimetres

ΔW = increment in load, in newtons

ΔS = increment of deflection at the mid-length of the test piece, corresponding to ΔW

All the calculated MOE values were then recorded for analysis.

Modulus of Rupture (MOR) Testing

The testing method for MOR was exactly the same as for MOE testing. Equation 5.24 shows the calculation of test piece's bending strength:

$$MOR = \frac{3WL}{2bt^2} \qquad \text{Equation 5.24}$$

where,

MOR = Modulus of Rupture, in megapascals

W = Ultimate failure load, in newtons

L = distance between the centre of the supports, in millimetres

b = width of the test piece, in millimetres

t = thickness of test piece, in millimetres

The calculated MOR values were recorded for further analysis.

Density Testing

Density was determined as the ratio of the mass of each test piece to its volume. These results were used to estimate the density of whole panels.

A micrometer was used for thickness measurement and for length and width measurement, a sliding calliper was used. A balance allowing measurements to 0.01g was used for weight measurement. Similar to the IB testing, five pieces of 50 x 50 mm test samples were prepared and conditioned.

Each test piece was first weighed to an accuracy of 0.01g. The dimensions of test pieces could easily obtained by measuring the thickness and width. The density (ρ) of each test piece was calculated using Equation 5.25.

$$\rho = \frac{m}{v} \times 10^6 \qquad \text{Equation 5.25}$$

where,

ρ = density, in kilograms per cubic metre

m = mass of test pieces, in grams

v = volume of test piece, in cubic millimetres

The density of panels was obtained by calculating the arithmetic mean of the densities of all the test pieces taken from the same panel.

5.6 Summary

This chapter has introduced the procedure developed in the RMIT laboratory for processing the materials and manufacturing particleboards. The testing equipments and procedures have been presented in detail. The mechanical and physical properties were found in accordance the testing methods specified by the AS/NZS standards.

The manufacturing process was tested by manufacturing a softwood board using similar proportions and processes to those adopted in the industry. The process developed at RMIT University was observed to provide a board with a similar quality to commercial boards.

In all subsequent work, the manufacturing process was maintained, except where specific changes were needed to suit the planned series of experiments. These changes are mentioned, where applicable.

Chapter 6

PARTICLEBOARD PRODUCTION USING MIXTURES OF SOFTWOOD AND HARDWOOD RESIDUE: SERIES 1

6.1 Overview

After a detailed literature review, the processes for the production of particleboard and the testing of major mechanical properties were established, as presented in earlier chapters. This chapter presents the results of screening tests of particleboard made from mixtures of softwood and hardwood residues. Process and material variables were identified for the experimental work. A number of boards were made and the results were analysed to obtain the significant factors affecting board properties.

6.2 Introduction

Based on previous research done at RMIT University (Nirdosha,2007), the basic method to produce particleboard from hardwood residues in the laboratory has been established. This work has verified that particleboard made in the laboratory has acceptable mechanical properties. However, the board product developed requires higher resin consumption and has a higher board density than industrial particleboard. It also requires a longer pressing time to produce. This chapter describes the first experimental study of particleboard using mixtures

of softwood and hardwood residues in order to optimize board properties and the production process.

The effects of material and process variables on the properties of particleboard are then presented based on the results of testing. The most significant variable on each property is reported, and the results are compared with the results of particleboard from hardwood residues.

6.3 Screening Test 1: Identification of significant variables

Eight material and process variables were identified for Screening Test 1. A multivariate factorial experimental design was carried out using identified variables with two levels (as shown in Table 6.1) to investigate the effects on the mechanical and physical properties of the finished boards. All these variables were selected based on the findings of the literature review and discussion with specialists in the particleboard industry (Justin Tonks from D & R Henderson).

The process variables considered are the pressing time and temperature. The material variables are the surface and core moisture content, surface and core resin content, and hardwood proportions in surface and core. In comparison with Nirdosha et. al (2005), the surface moisture content range set was comparatively low at 9 % to 14 % compared to the 11 % to 22 % in the earlier work.

Variables	**Low Value**	**High Value**	**Units**
Core Moisture Content (A)	7	13	% of dry wt of the board
Surface Moisture Content (B)	9	14	% of dry wt of the board
Core Resin Content (C)	8	16	Resin load as a % of dry wt of the board
Surface Resin Content (D)	8	16	Resin load as a % of dry wt of the board
Hardwood portion in Core (E)	40	60	% of total particles wt in core
Hardwood portion in Surface (F)	40	60	% of total particles wt in surface
Pressing Temperature	150	200	^{o}C

(G)			
Pressing Time (H)	120	240	Sec

Table 6.1 Variables used in the experimental design for three-layer particleboard

The target density of the board was maintained at 680 kg/m^3 at thicknesses between 13 to 22 mm. Table 6.2 below shows the typical or target property values for AS/NZS standard particleboard.

Property	**Units**	**Thickness Class – mm (13 – 22 mm)**
Density	Kg/m^3	660 – 680
Bending Strength (MOR)	MPa	15
Bending Stiffness (MOE)	MPa	2600
Internal Bond Strength (IB)	kPa	450

Table 6.2 Typical or target properties values for standard particleboard

An experimental screening series, obtained from a 1/8 fractional (2^{8-3}) factorial design with one centre point, was carried out to investigate the effects of variables on the properties of the finished boards. 33 different experimental boards were produced in the laboratory, as shown in Table 6.3.

RunOrder	Moisture core	Moisture Surface	Resin Core	Resin Surface	Hardwood portion in core	Hardwood portion in surface	Press Temperature	Press time
1	13	14	16	16	60	60	200	240
2	7	9	8	16	40	40	200	120
3	13	14	16	8	40	60	150	240
4	7	14	8	16	40	60	150	240
5	7	14	8	16	60	60	150	120
6	7	14	16	16	60	40	150	240
7	7	9	8	8	40	40	150	240
8	13	14	16	8	60	60	150	120
9	13	14	8	16	60	40	200	120
10	7	9	16	16	40	60	200	240
11	13	9	16	8	40	40	200	120
12	13	14	8	8	40	40	150	120
13	13	9	16	8	60	40	200	240
14	13	9	16	16	60	40	150	120
15	13	14	8	8	60	40	150	240
16	13	9	8	16	40	60	150	120
17	13	9	16	16	40	40	150	240
18	13	9	8	16	60	60	150	240
19	7	9	8	8	60	40	150	120
20	7	9	16	8	60	60	150	240
21	7	14	8	8	60	60	200	240
22	13	14	8	16	40	40	200	240
23	13	9	8	8	40	60	200	240
24	7	9	16	8	40	60	150	120
25	10	11.5	12	12	50	50	175	180
26	7	9	16	16	60	60	200	120
27	7	14	8	8	40	60	200	120
28	7	14	16	8	60	40	200	120
29	7	9	8	16	60	40	200	240
30	7	14	16	8	40	40	200	240
31	13	14	16	16	40	60	200	120
32	7	14	16	16	40	40	150	120
33	13	9	8	8	60	60	200	120

Table 6.3 33 experimental boards from screening test

6.4 Materials and Methodology

6.4.1 Materials

A mixture of hardwood and softwood residues was utilised to produce particleboard in the laboratory. Each residue comes in two types: mulch and fine. Mixtures of mulch and fine in each layer of particleboard can obtain a better particle size distribution that is vital for better compaction (Nirdosha et al., 2005). According to Nirdosha et al. (2008), a mix of 65 % fine with 35 % mulch was used for the surface layer while a mix of 55% fine with 45 % mulch was used for the core layer. A similar approach was adopted in the work reported here.

On average, hardwood is of higher density and hardness than softwood. The particle size of hardwood residues is comparatively larger than softwood residues. By mixing hardwood and softwood, the particleboard produced was expected to have relatively lower density than the particleboard with 100 % hardwood residues. Urea formaldehyde (UF) was used as the resin for the production of the particleboard.

6.4.2 Methodology

The residues were first oven-dried at 105 ^{0}C for at least 24 hours to remove the initial surface moisture of the particles. Then, mulch and fine were sieved and measured separately according to the mix proportions calculated for each surface and core layer. After measuring, the mulch and fine of the hardwood and softwood residues were mixed in a mixer for approximately one minute prior to spraying with a mixture of water and resin. The resinated mix was then moulded into three layers in a mould of 300 mm x 400 mm. The mat was then inserted into a cold press and pressed to 20 mm thickness. After the cold pressing at a selected pressing time, the mat was then transferred to a hot press and compressed until it reached 15.2 mm hexagonal stoppers. The final boards were conditioned in a humidity chamber at a humidity of 65 % and temperature of 20 ^{0}C prior to testing as per AS/NZS 4266 (2004).

6.4.3 Test Procedures

After conditioning for a week, all the finished boards were trimmed and cut into sizes of 100 mm x 300 mm and 50 mm x 50 mm for testing. The 50 mm x 50 mm samples from each board were used to measure the density and internal bond strength (IB) according to AS/NZS 4266.6 (2004). The density was simply measured using the weight/volume method. The 100 mm x 300 mm samples were used to test the Modulus of Elasticity (MOE) and Modulus of Rupture (MOR) in accordance with AS/NZS 4266.5 (2004). Results were then compared to the target properties.

6.5 Results and Discussion

All boards were tested and the results obtained are given in Table 6.4. MINITAB 15 was used to analyse the results based on the theories of experimental design to calculate the test statistical values (T) and the probability of null hypothesis (P) of each variable, as shown in Table 6.5. A variable with a significant effect will have a higher 'T' value than those non-significant variables (Myers and Montgomery, 2002). The 'P' value indicates the level of significance of the variable. If $P<0.05$ for any variable with respect to any board property, that variable has a significant effect on the board property with 95 % significance level.

Board no.	Moisture core	Moisture Surface	Resin Core	Resin Surface	Hardwood portion in core	Hardwood portion in surface	Press Temperature	Press time	MOE	MOR	Density	IB
1	13	14	16	16	60	60	200	240	883.48	13.26	651.36	117.00
2	7	9	8	16	40	40	200	120	1483.44	12.55	675.61	642.12
3	13	14	16	8	40	60	150	240	912.58	10.80	599.58	311.38
4	7	14	8	16	40	60	150	240	657.86	2.80	705.68	232.12
5	7	14	8	16	60	60	150	120	554.80	5.14	588.57	154.53
6	7	14	16	16	60	40	150	240	971.35	8.10	711.06	464.00
7	7	9	8	8	40	40	150	240	720.51	3.74	623.19	110.25
8	13	14	16	8	60	60	150	120	706.87	5.76	607.59	180.00
9	13	14	8	16	60	40	200	120	795.16	5.45	767.09	168.75
10	7	9	16	16	40	60	200	240	1084.14	10.75	640.84	533.75
11	13	9	16	8	40	40	200	120	757.47	6.39	753.25	512.38
12	13	14	8	8	40	40	150	120	518.67	4.52	657.60	104.50
13	13	9	16	8	60	40	200	240	1296.34	9.97	758.35	286.38
14	13	9	16	16	60	40	150	120	347.57	4.21	667.51	173.00
15	13	14	8	8	60	40	150	240	686.94	5.92	746.50	206.30
16	13	9	8	16	40	60	150	120	607.10	4.05	761.75	292.62
17	13	9	16	16	40	40	150	240	1172.13	8.75	663.16	349.75
18	13	9	8	16	60	60	150	240	1453.66	10.74	659.20	390.00
19	7	9	8	8	60	40	150	120	674.76	5.48	648.05	179.88
20	7	9	16	8	60	60	150	240	815.06	7.09	608.22	246.62
21	7	14	8	8	60	60	200	240	1240.00	12.05	642.59	435.44
22	13	14	8	16	40	40	200	240	1267.75	8.96	672.71	416.75
23	13	9	8	8	40	60	200	240	283.44	4.60	593.54	263.29
24	7	9	16	8	40	60	150	120	878.46	8.41	665.75	138.25
25	10	11.5	12	12	50	50	175	180	1170.73	9.56	633.75	141.38
26	7	9	16	16	60	60	200	120	1730.46	14.98	728.74	329.38
27	7	14	8	8	40	60	200	120	1329.82	11.98	686.32	344.50
28	7	14	16	8	60	40	200	120	301.59	2.42	632.04	304.75
29	7	9	8	16	60	40	200	240	979.53	6.77	650.82	454.50
30	7	14	16	8	40	40	200	240	782.96	7.58	732.16	267.50
31	13	14	16	16	40	60	200	120	905.82	4.67	704.40	313.50
32	7	14	16	16	40	40	150	120	992.06	6.13	766.13	420.53
33	13	9	8	8	60	60	200	120	988.91	8.84	667.54	386.10

Table 6.4 Experimental variables and results

Term	MOR		IB		MOE	
	T	P	T	P	T	P
Moisture core	-1.79	0.133	-30	0.001	-3.56	0.016
Moisture Surface	-2.33	0.067	-32.3	0.001	-3.9	0.011
Resin Core	3.09	0.027	6.35	0.024	0.65	0.542
Resin Surface	2.32	0.068	44.81	0	6.61	0.001
Hardwood portion in core	1.88	0.119	-29.62	0.001	0.16	0.879
Hardwood portion in surface	5.73	0.002	-14.98	0.004	2.84	0.036
Press Temperature	7.82	0.001	69.51	0	7.6	0.001
Press time	4.12	0.009	16.79	0.004	3.61	0.015
Moisture core*Moisture Surface	3.04	0.029	-31.43	0.001	2.89	0.034
Moisture core*Resin Core	NS	NS	-5.2	0.035	NS	NS
Moisture core*Resin Surface	NS	NS	-47.02	0	NS	NS
Moisture core*Hardwood portion in core	2.63	0.047	-20.47	0.002	3.08	0.027
Moisture core*Hardwood portion in surface	-2.34	0.066	17.4	0.003	-3.28	0.022
Moisture core*Press Temperature	-4.89	0.005	-34.68	0.001	-4.18	0.009
Moisture core*Press Time	7.37	0.001	NS	NS	6.67	0.001
Moisture Surface*Hardwood portion in core	-1.61	0.168	0.63	0.593	-5.58	0.003
Moisture Surface*Press time	5.11	0.004	18.26	0.003	2.12	0.087
Resin Core*Resin Surface	2.58	0.049	-10.2	0.009	NS	NS
Resin Core*Hardwood portion in core	NS	NS	-27.28	0.001	-2.07	0.093
Resin Core*Press Temperature	5.08	0.004	-40.44	0.001	-3.42	0.019
Resin Surface*Hardwood portion in core	2.07	0.094	-42.85	0.001	-2.17	0.083
Resin Surface*Press time	NS	NS	18.56	0.003	NS	NS
Hardwood portion in core*Hardwood portion in surface	5.94	0.002	15.1	0.004	7.41	0.001
Hardwood portion in core*Press Temperature	0.6	0.578	-32.28	0.001	NS	NS
Hardwood portion in core*Press time	4.41	0.007	38.43	0.001	6.22	0.002
Hardwood portion in surface*Press time	NS	NS	13.01	0.006	-5.25	0.003
Press Temperature*Press time	-1.5	0.194	-34.1	0.001	-5.71	0.002
Moisture core*Moisture Surface*Hardwood portion in core	-1.75	0.14	-22.87	0.002	-2.36	0.064
Moisture core*Moisture Surface*Press time	-1.97	0.105	9.16	0.012	-5.13	0.004
Moisture core*Resin Core*Resin Surface	-5.48	0.003	-38.99	0.001	-9.49	0

Table 6.5 Estimated effects and coefficients for physical properties of a board

6.5.1 Factors affecting the mechanical and physical properties of particleboard

Table 6.4 shows the 33 different recipes produced by experimental design to produce particleboards. Particleboard no. 26 has the highest MOE at 1730.457 MPa, 14.977 MPa (MOR) and 329.376 kPa (IB). According to Table 6.2, these optimal boards do not satisfy the requirements for the mechanical properties of standard particleboard.

Table 6.5 indicates that pressing temperature and pressing time significantly influence the properties of particleboard. Karr et al., (1999) found that pressing temperature and pressing time are important factors for particleboard as they provide the heat and time for the resin curing and create more cross-linking sites. Because the entire board is not uniformly heated throughout the thickness, the curing of the resin does not occur uniformly; the surface layer is cured first and then curing occurs in the core through heat transfer from the surface layer. Therefore, the core layer will always be at the lowest temperature and it might need sufficient pressing time to reach a significant temperature to allow the resin to cure.

The hardwood proportion in the surface is one of the critical variables that affect the properties of particleboard. Since the bending strength is mainly dependent on the surface layer, the hardwood portion in the surface has more effect (positive) on MOR and MOE. This can be explained by the particle size effect. Post (1958) found that particle geometry was much more significant than adhesive content. Various attempts have been made to investigate the effect of particle size on particleboard properties (Turner 1954; Post 1958; Brumbaugh 1960; Heebink and Hann 1959; Lehmann and Geimer 1974). According to these studies, the strength properties of particleboard increase when the particle size increases. Therefore, the optimal bending strength properties were obtained when the hardwood portion in the surface is high at 60 %, which contained more hardwood particles that are relatively larger than the softwood particles.

The resin surface is also a significant factor for particleboard properties. The amount of resin is important to create proper bonding between particles. Generally in-plane and lateral bending loads are primarily resisted by the surface materials in a structural element (Vinson 1999). Hence, having higher amounts of surface resin in the surface layer is vital to create inter-particle bonding in the surface layer.

The moisture in the core combined with pressing time has a significant effect on particleboard. Core moisture and pressing time directly influence the curing of the resin in the core. With a higher core moisture content and insufficient pressing time, excess moisture may be trapped in the board which creates a weaker bonding and increased thickness swelling. It has been explained by previous researchers (Beech, 1975; Suchsland, 1969; Smith, 1982) that as releasing the pressure after hot pressing, higher moisture content excess in the core may lead to high spring-back and non-reversible excessive dimensional changes of the board.

6.5.2 Comparison of results with particleboard from hardwood residues

In order to lower the density of the hardwood particleboard, mixtures of hardwood and softwood residues were utilised in proportions of 40 % or 60 % of hardwood within the surface and core layers. Surface moisture content, surface resin content and pressing time were also lowered in order to improve the deficiencies of hardwood in particleboard production without affecting the properties.

However, the outcomes of this first experimental program did not confirm the hypothesis that use of softwood as part furnish would improve the board properties and processing parameters. Approximately 8 boards out of 33 boards had a mean density within the target density range. The variation of the target density was a result of thickness swelling of the board, caused by the interaction of the processing variables. This indicates that mixtures of hardwood and softwood do not have a significant effect on board density.

The highest MOR and MOE values of particleboard made of mixtures of hardwood and softwood were comparatively lower than the values for hardwood residue particleboard. Softwood/hardwood boards had mechanical properties of 1730.457 MPa (MOE) and 14.977 MPa (MOR) compared to 2088 MPa (MOE) and 16.59 (MOR) of the hardwood boards (Nirdosha, 2007). It is hypothesised that the lower surface moisture content and surface resin content directly influenced the properties of the board. Nirdosha, (2007) stated that MOE and MOR are mainly dependent on the surface layer of the board. Therefore, the difference of surface moisture content and surface resin content will affect the properties of the boards. According to Lehmann (1960), an increase in surface moisture content from low to high

increases the MOR and MOE. As observed from Table 6.1, the surface moisture content and core moisture content were set in a very similar range, which may be considered as uniform moisture content. Heebink (1974) observed improved board strength when he used non-uniform moisture content (15 % for surface and 5 % for core). Therefore, the strength of the hardwood particleboard with non-uniform moisture content (22 % for surface and 15 % for core) is relatively higher. Mixtures of hardwood and softwood residues also affected the MOR value. This is due to the particle size of the board furnish. The hardwood particleboards were produced from only hardwood residues which are relatively larger than the softwood residues. According to a previous investigation (Lehmann and Geimer 1974), increased flake length up to 5 cm (particle size) increases the MOR. The particle sizes in the boards produced from mixtures of hardwood and softwood residues were smaller and hence a lower MOR value was obtained.

6.4 Summary

Results of the laboratory studies indicated that the following variables are significant in influencing the mechanical properties of particleboard made of mixtures of hardwood residue and softwood flakes:

- Press temperature and pressing time. These variables assist in resin curing and enable stronger bonding within the particles.
- Particle size and consequently the hardwood proportion in the surface. A higher hardwood proportion in the surface results in optimal strength particleboard.
- Moisture surface and moisture core. The high moisture content in the surface aids conductivity and heat transfer to cure the resin

In comparison to hardwood particleboard, particleboard produced from mixtures of hardwood and softwood residues has lower optimal strength properties. However, the board density obtained remains higher than the target density. Adjustment of the range of variables range is essential to achieve adequate properties. The second series of experiments was planned based on the analysis of the screening tests.

Chapter 7

PARTICLEBOARD PRODUCTION USING MIXTURES OF SOFTWOOD AND HARDWOOD RESIDUES IN THE CORE AND HARDWOOD IN THE SURFACE: SERIES 2

7.1 Introduction

As discussed in Chapter 6, a mixture of hardwood and softwood residues was utilised in proportions of 40 % or 60 % of hardwood within the surface and core layers in order to lower the density of the hardwood particleboard. Surface moisture content, surface resin content and pressing time were been lowered in order to overcome the deficiencies of the hardwood particleboard production process without affecting the properties of the board.

However, the outcomes of the first series of experimental program did not confirm the hypothesis that use of softwood as part of the furnish would improve board properties and processing parameters. Particleboard produced from mixtures of hardwood and softwood residues had lower optimal strength properties, and the density obtained remained higher than the target density. Adjustment of the range of variables was therefore identified as essential to achieve adequate properties.

7.2 Hypothesis

As reported in Chapter 6, results of the laboratory studies indicated that the following variables are significant in influencing the mechanical properties of particleboard made of mixtures of hardwood residue and softwood flakes.

- Press temperature and pressing time. These assist in resin curing and enable stronger bonding within the particles.
- Particle size and consequently the hardwood proportion in the surface. The higher hardwood proportion in the surface results in an optimal strength of particleboard.
- Moisture surface and moisture core. High moisture content in the surface aids conductivity and heat transfer to cure the resin.

It appears that some alterations to these variables are essential.

7.2.1 Hypothesis 1: Hardwood particles only in surface layer will improve bending properties of particleboard

Many reports have appeared concerned with the effect of particle geometry and alignment on particleboard's strength properties. Post (1958) found a continuous increase in bending strength for oak particleboard with increasing flake length over the studied range of 0.5 to 4 inches. However, as the flake thickness increased above 0.01 inch, the properties decreased for all flake lengths. This indicates that particle thickness has more influence on the bending strength of particleboard than particle length. Larger particles can be satisfactorily bonded at lower resin contents as the particle surface area is drastically reduced as the particle size increases. Gertjejansen and Haygreen (1973) found that substantial adhesive savings are possible with the larger wafer, provided that other properties are maintained.

The results reported in Chapter 6 show that higher hardwood proportion in the surface result in higher strength particleboard. It appears that larger particles in the surface assist improved particleboard strength. As seen in Figure 5.2 and Figure 5.3, hardwood residues are relatively larger than softwood residues. Hence, it was decided to utilise hardwood particles only in the surface layer in Series 2 experiments in order to improve bending strength.

7.2.2 Hypothesis 2: Non-uniform moisture distribution over surface and core layer will improve board properties

Table 6.1 shows that the moisture content in surface and core layers are set in a very similar range, which may be considered uniform moisture distribution. Uniform moisture distribution might result in excessive moisture which requires additional pressing time to exit through the edges of the board. Delamination or spring-back of the board may occur with insufficient pressing time and excess moisture. However, additional pressing time for the removal of excess moisture will cause over-curing of the surface layer which is in direct contact with the hot platen. Therefore, non-uniform moisture distribution over surface and core layers was applied in order to retain the advantage of rapid heat transfer to the core. With higher levels of moisture at the surfaces than at the core, rapid heat transfer occurs without unduly lengthening the pressing time. Heebink (1974) observed improved board strength when non-uniform moisture contents (15 % for surface and 5 % for core) were applied instead of using uniform moisture content (12 %) for three-layer particleboard.

7.3 Screening test 2: Identification of significant variables

Against hypothesis as discussed, only hardwood residues are utilised in the surface layer of particleboard. Non-uniform moisture content was set for the surface and core layers, and there was a mixture of hardwood and softwood in the core layer. Seven material and process variables were identified for screening test 2 as shown in Table 7.1. Moisture content for the surface (A), moisture content for the core (B), resin load for the surface (C), resin load for the core (D), hardwood portion in core (E), pressing time (F), and pressing temperature (G) were the seven experimental variables identified.

Variables	Low Value	High Value	Units
Surface Moisture Content (A)	11	20	% of dry wt of the board
Core Moisture Content (B)	5	12	% of dry wt of the board
Surface Resin Content (C)	8	16	Resin load as a % of dry wt of the board
Core Resin Content (D)	7	11	Resin load as a % of dry wt of the board
Hardwood portion in Core (E)	20	80	% of total particles wt in surface
Pressing Time (F)	180	300	Sec
Pressing Temperature (G)	150	200	^{o}C

Table 7.1 Variables used in the experimental design for three-layer particleboard

Some manipulations were carried out on these variables. Nirdosha et. al (2005) identified that higher moisture content may be required for hardwood particleboard compared to softwood particleboard. In the softwood particleboard industry, approximately 11 % to 15 % moisture content is used in the surface layer, while 8 % to 10 % is used for the core.

7.4 Experimental design

A screening experimental series was conducted to identify the most significant parameters and their interactions controlling the properties of three-layer variables made with mixtures of hardwood and softwood residues. As Table 7.1 shows, seven variables were used to obtain the screening experimental series at 1/8 fractional (2^{7-3}) factorial design. 16 different experimental boards were produced in the laboratory with one replicate. Different treatment combinations with respect to this 2^{7-3} design are presented in Table 7.2.

RunOrder	Core MC	Surface MC	Core Resin	Surface Resin	Hardwood Portion in Core	Pressing Temperature	Pressing Time
1	5	11	11	8	80	200	300
2	5	20	11	8	20	150	300
3	12	20	7	8	20	200	300
4	12	20	11	16	80	200	300
5	12	20	11	8	80	150	180
6	12	11	7	16	80	200	180
7	12	11	11	16	20	150	300
8	5	11	7	8	20	150	180
9	5	20	7	8	80	200	180
10	12	20	7	16	20	150	180
11	5	20	11	16	20	200	180
12	5	11	7	16	20	200	300
13	5	11	11	16	80	150	180
14	12	11	11	8	20	200	180
15	5	20	7	16	80	150	300
16	12	11	7	8	80	150	300

Table 7.2 Different treatment combinations with respect to 2^{7-3}

7.5 Materials and methodology

As for particleboard production discussed in Chapter 6.3.1, the same materials were used to prepare three-layer particleboard, the only difference being the content of the surface layers. Against the hypothesis 1, only hardwood residues were applied in the surface layers of this screening experimental series.

The laboratory procedure for the particleboard manufacturing has been detailed in Chapter 6.3.2. All the completed boards were then sampled, conditioned and tested for their physical and mechanical properties.

As described in Section 6.3.3, the finished three-layer boards were then tested in accordance with AS/NZS. Samples of 50 mm x 50 mm were prepared from each board for IB testing, while MOR and MOE were tested using the samples of 100 mm x 300 mm. The mean density of each board was also measured.

7.6 Results and discussions

Table 7.3 shows the result of 16 different recipes given by experimental design to manufacture particleboards. MOE, MOR, IB and density were four major features tested.

RunOrder	Core MC	Surface MC	Core Resin	Surface Resin	Hardwood Portion in Core	Pressing Temperature	Pressing Time	MOE	MOR	IB	Density
1	5	11	11	8	80	200	300	1292.855	7.055638	308	679.3301
2	5	20	11	8	20	150	300	1546.595	7.652659	296.748	693.9952
3	12	20	7	8	20	200	300	1767.59	8.153198	235.5	665.4026
4	12	20	11	16	80	200	300	2178.931	10.46422	780	732.7461
5	12	20	11	8	80	150	180	835.3172	6.220213	361.376	675.2475
6	12	11	7	16	80	200	180	1809.632	12.75417	403.5	661.604
7	12	11	11	16	20	150	300	1495.572	6.14041	77.5	666.7078
8	5	11	7	8	20	150	180	1098.737	3.807867	349.624	641.3527
9	5	20	7	8	80	200	180	1189.296	6.525364	410.376	658.0952
10	12	20	7	16	20	150	180	1461.894	9.04152	55.376	621.1874
11	5	20	11	16	20	200	180	1675.808	12.7195	554.876	743.789
12	5	11	7	16	20	200	300	1182.132	4.71134	160.624	584.5161
13	5	11	11	16	80	150	180	856.1715	5.339518	605.5	612.5286
14	12	11	11	8	20	200	180	828.3297	5.805189	149.376	595.2863
15	5	20	7	16	80	150	300	1575.355	4.884012	310.876	597.9741
16	12	11	7	8	80	150	300	966.5961	4.222908	378.748	604.6493

Table 7.3 Experimental variables and results

The test results in the table above show that particleboard no. 4 has the optimal board properties with MOE at 2178.931 MPa, MOR at 10.464 MPa, IB at 780 kPa and density at 732.746 kg/m^3. According to AS/NZS standards, none of these boards satisfy the minimum requirements of standard particleboard as stated in Table 6.2. The optimal board requires extra resin and moisture with a longer pressing time. The overall particleboard density remains higher than the target density.

The results were then analysed using the theories of experimental design explained in Chapter 6. Variables with significant effects will have a non-zero mean, which would not fall along a straight line on a normal probability plot (Myers and Montgomery 2002). Table 7.4 shows the test statistical values (T) and the probability of null hypothesis (P) of each variable. Effects with higher 'T' value are considered significant variables, while the 'P' value indicates the level of significance of the variable. If $P < 0.05$ for any variable with respect to any board property, the null hypothesis is not true and the factor affects the board property with 95 % significance.

Terms	MOE		MOR		IB	
	T	P	T	P	T	P
Core MC	2.34	0.257	20.05	0.032	-7.33	0.086
Surface MC	6.82	0.093	31.4	0.02	7.55	0.084
Core Resin	-0.86	0.547	14.48	0.044	10.94	0.058
Surface Resin	6.84	0.092	32.96	0.019	6.05	0.104
Hardwood Portion in Core	-0.89	0.537	-1.12	0.463	22.16	0.029
Pressing Temperature	5.27	0.119	41.43	0.015	7.48	0.085
Pressing Time	5.68	0.111	-17.72	0.036	-4.51	0.139
Core MC*Surface MC	-1.04	0.486	-11.73	0.054	3.62	0.172
Core MC*Core Resin	-2.51	0.242	-36.47	0.017	-3.15	0.196
Core MC*Surface Resin	6.02	0.105	22.59	0.028	NS	NS
Core MC*Hardwood Portion in Core	2.09	0.285	19.06	0.033	14.96	0.043
Core MC* Pressing Temperature	3.94	0.158	4.41	0.142	10.88	0.058
Core MC* Pressing Time	1.76	0.329	-1.49	0.376	17.77	0.036
Surface MC*Surface Resin	NS	NS	NS	NS	4.43	0.141
Core MC*Surface MC*Surface Resin	-3.38	0.183	-15.82	0.04	3.11	0.198

*NS = Not Significant

Table 7.4 Estimated Effects and Coefficients for properties of a board

Normal probability plots and Pareto charts were used to analyse the data to identify significant variables as well as to find the level of significance.

7.6.1 Factors affecting Modulus of Elasticity (MOE) of particleboard

The T and P values for each process variables with respect to MOE are displayed in Table 7.4. The most significance parameters affecting MOE can be obtained from the table with P <

0.05. The higher the T value, the higher the effect of the variable on MOE. The normal probability plot for standardized effect for MOE (Figure 7.1) was used to verify the significant variables.

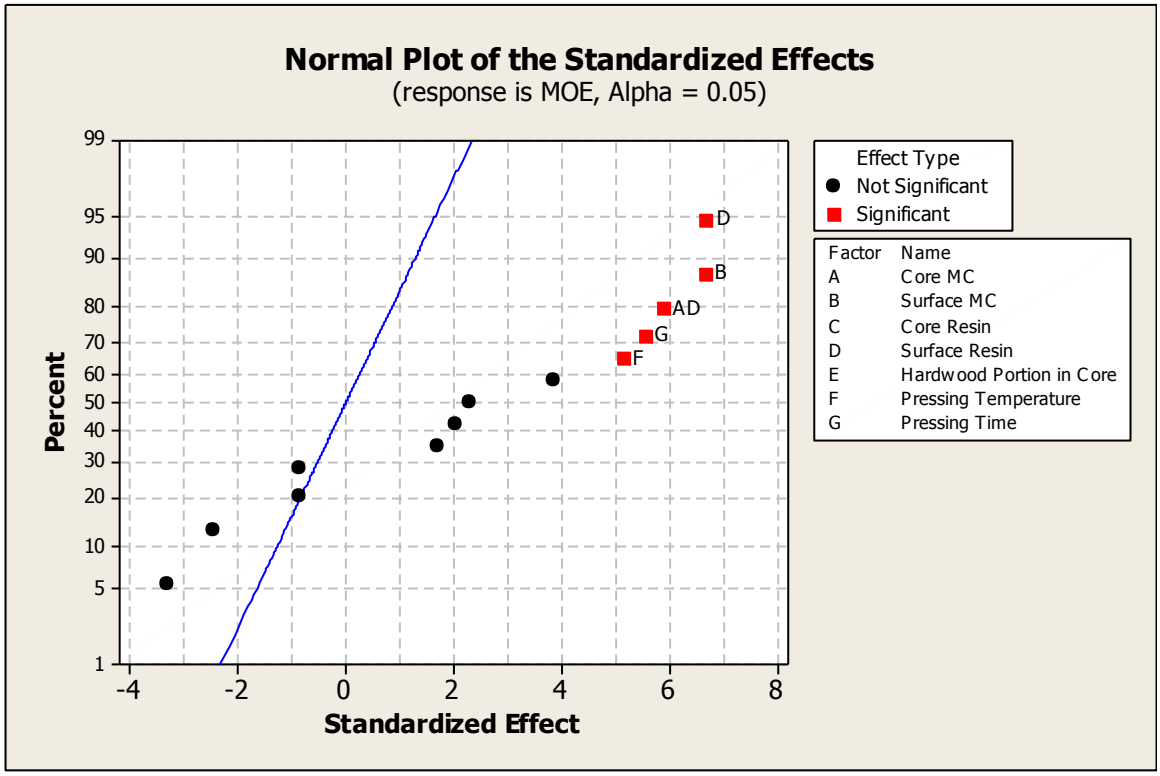


Figure 7.1 Normal Probability plot for standardized effect for MOE

Significant parameters which control MOE are marked with red squares. The test statistical value and the effect that satisfies the 95 % significance margin value is around (+ or -) 2.4. The blue line in Figure 7.1 indicates the two positive and negative margins of the test statistical values. Effects or combinations of effects which negatively influence MOE will remain on the negative side of the plot. Figure 7.1 shows that surface resin content, surface moisture content, combination of core moisture content and surface resin content, pressing time and pressing temperature have positive significant effects on MOE.

Figure 7.2 shows the Pareto chart which highlights the significant variables with respect to MOE. This figure shows that surface resin content has the most significant effect on the MOE. Resin is the major binding agent to create bonding between particles in particleboard production. Vinson (1999) stated that lateral bending loads are primarily resisted by the surface materials in a structural element. Several researchers reported in Chapter 3 found improvement in board strength by increasing the adhesive level. Hence, having a higher

amount of surface resin content is vital to create inter-particle bonding in the surface layer, which creates a stronger surface layer that directly influences the MOE value of particleboard.

Surface moisture content has the second highest effect on MOE. Surface moisture content directly influences the heat and mass transfer to the core. The increase in surface moisture content reduces the hardness of the wood mat and provides better compaction of the mat before resin curing. However, with a continuous increase in surface moisture above optimum levels, the MOE starts reducing. This is due to the excessive moisture migration to the core, which requires excessively longer pressing time to allow the removal of moisture through the edges to prevent delamination of the board.

The combination of core moisture content and surface resin content is the third significant variable for MOE. As discussed in the literature, moisture content is the major medium for heat transfer in particleboard. Therefore, in order to obtain adequate resin curing over the thickness of the particleboard, core moisture content must be maintained at a precise level to prevent excessive moisture within the board. The importance of surface resin content has been explained earlier in Chapter 3.

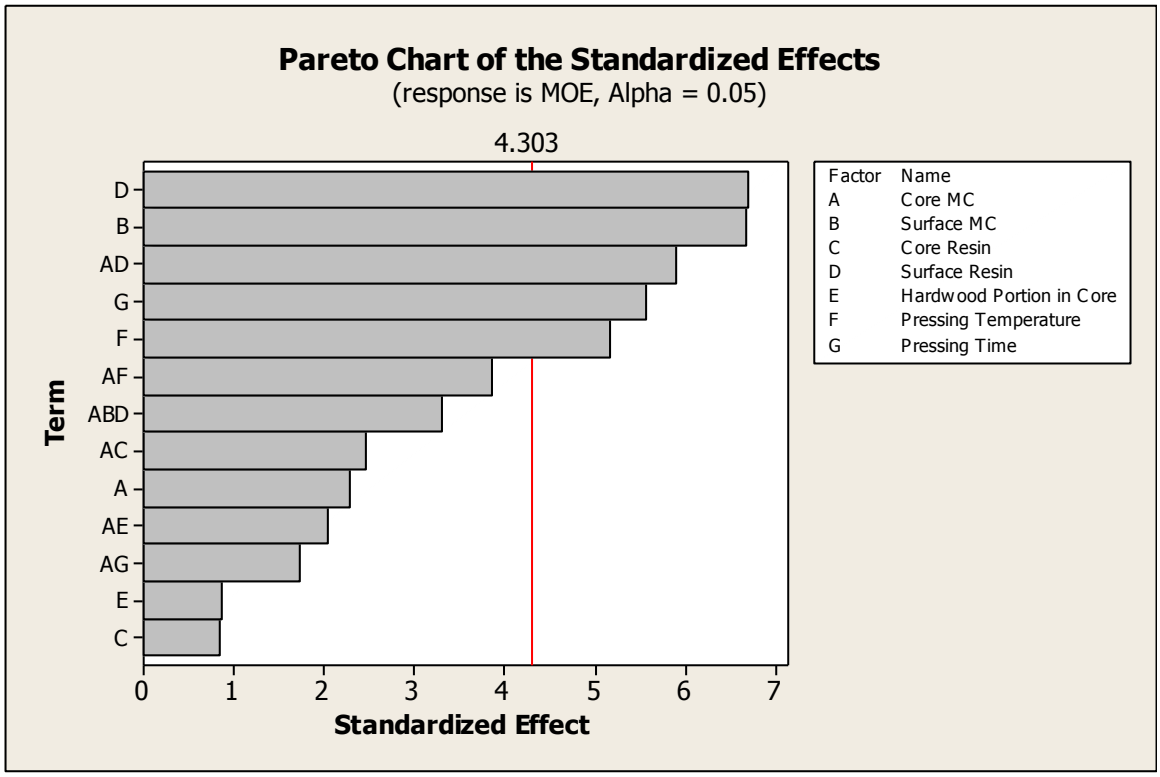


Figure 7.2 Pareto chart for standardized effect MOE

Pressing time and pressing temperature are also important for MOE. Pressing time and temperature are extremely important parameters in particleboard manufacturing. Both arerelated and have to be carefully controlled to ensure that the core temperature attains the level required to cure the adhesive without subjecting the board surface to a high, degradative temperature. As the entire board is not uniformly heated throughout the thickness, the curing of the adhesive may not occur uniformly. Since the heat transfer happens from the surface to the core layers, the core is always at the lowest temperature. Therefore, the pressing time and temperature have to be adequate to ensure that the core reaches a sufficiently high temperature to allow resin curing.

These individual factor effects or combinations of factor effects were further studied using contour plots. Contour plots show the behaviour of the MOE with respect to two variables. In contour plots, change in the response with respect to change in two variables (whilst others are kept constant) is presented. This plot is useful for establishing the desirable response values at the favourable variable settings found in the plot.

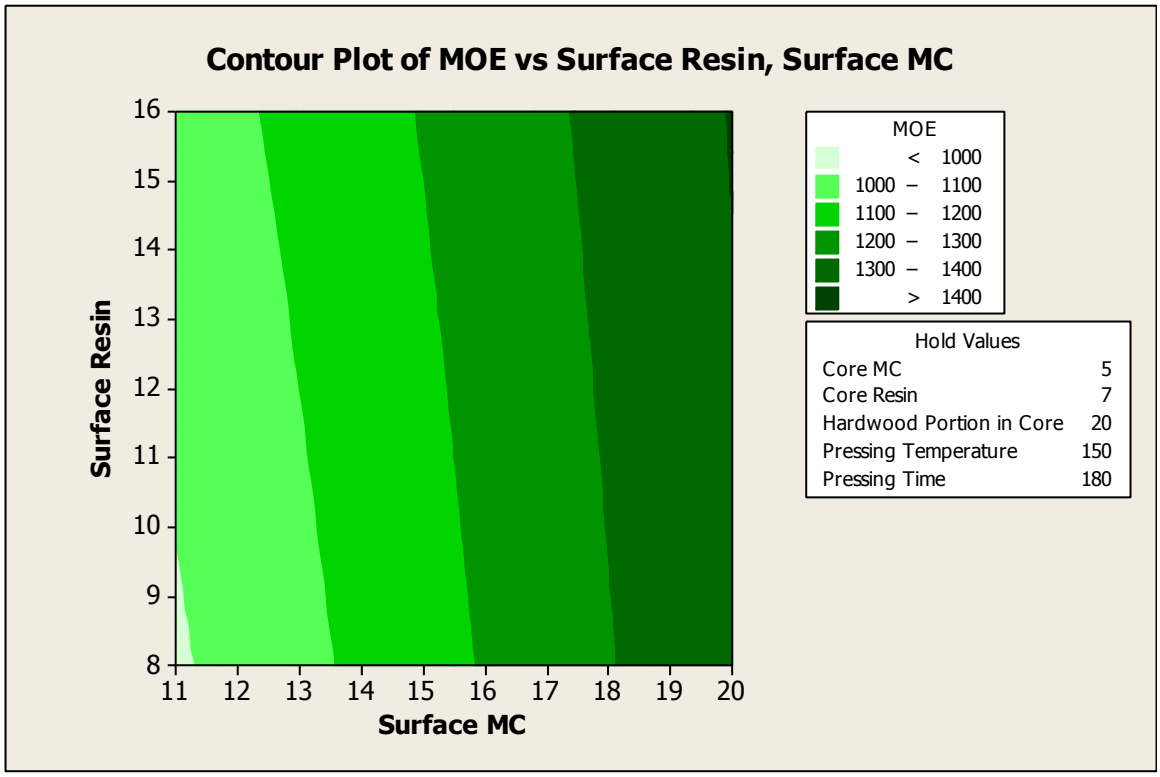


Figure 7.3 Contour Plot of MOE with respect to surface moisture and surface resin content

Figure 7.3 clearly shows the relationship between surface resin and surface moisture content and MOE. Surface moisture content is identified as a dominant factor that affects MOE. This

indicates that increasing surface MC by keeping surface resin constant leads to a steady increase of MOE. However, the highest MOE value can only be achieved at higher surface resin and surface MC, as indicated by the darkest green area.

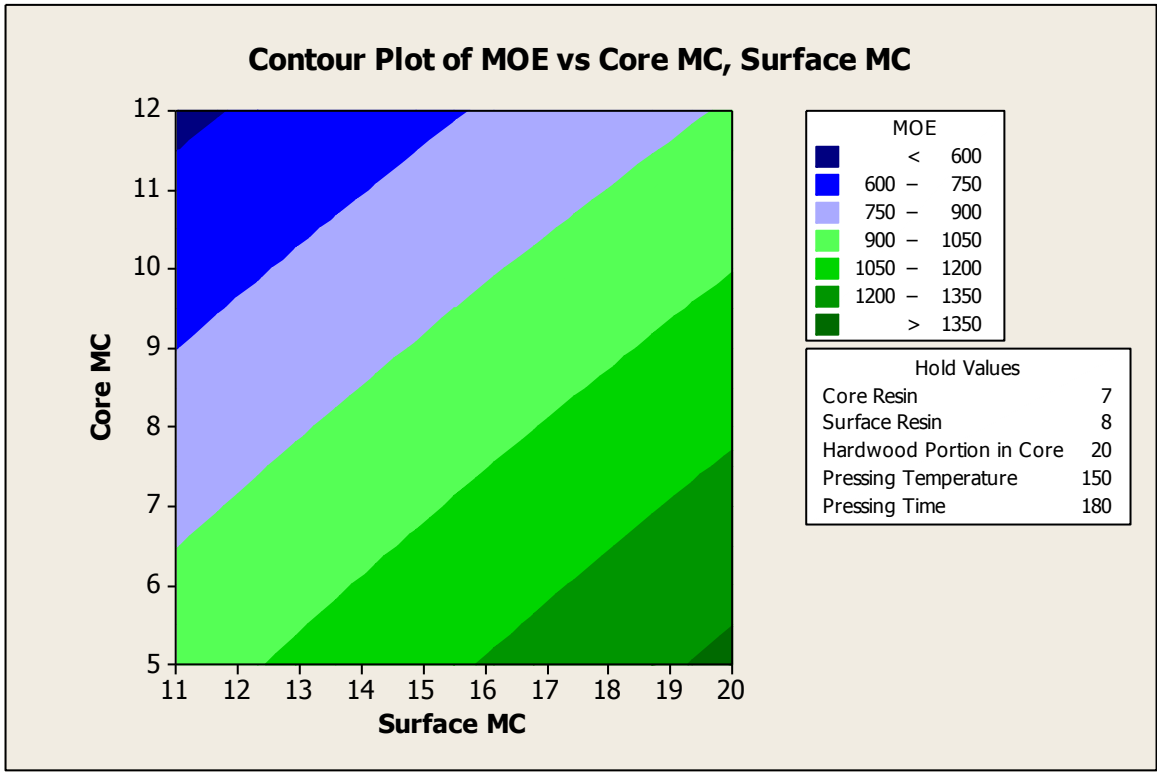


Figure 7.4 Contour plot of MOE with respect to Core MC and Surface MC

Figure 7.4 presents the contour plot of MOE when core MC and surface MC are changed. Hypothesis 2 can be verified in this plot, which shows that particleboard strength improves when there is non-uniform moisture content distribution over surface and core layers. Consistent with the findings of previous researchers, at lower core moisture content with higher surface moisture content, the highest possible MOE is achieved.

Figure 7.5 below shows the relationship between pressing time and pressing temperature and MOE. At short pressing times and low press temperatures, the MOE value is relatively poor, which may be attributed to too low hot press temperature and too short hot press time to produce consolidated panels. In particular, UF resin cannot produce consolidated panels if the pressing conditions do not reach the necessary curing conditions. When the pressing time is too short, the compaction of wood particles is inadequate to enable sufficient contact between particles and adhesive. With an increase in time and temperature, MOE value increases. It is also observed that pressing time and pressing temperature are inversely correlated. At a

higher pressing temperature, a shorter pressing time is needed to reach a satisfactory resin curing, which will lead to a high MOE. The dark green area at which high MOE can be obtained exists at only pressing times > 275 sec and pressing temperature > 165^{o}C.

Contour plot of MOE with respect to core MC and core resin is presented in Figure 7.6. Obviously, core moisture content has to be maintained at lower levels for high MOE. At low core MC, high MOE can be achieved at any level of core resin above 8 %.

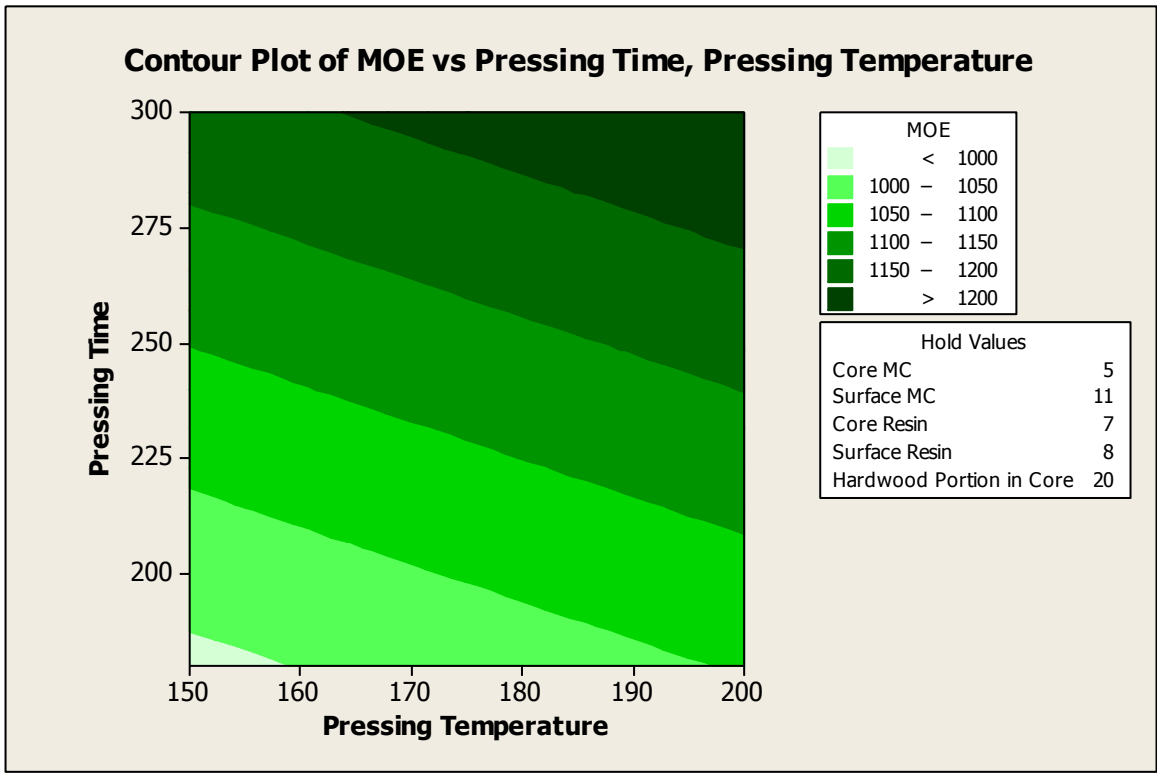


Figure 7.5 Contour Plot of MOE with respect to pressing time and pressing temperature

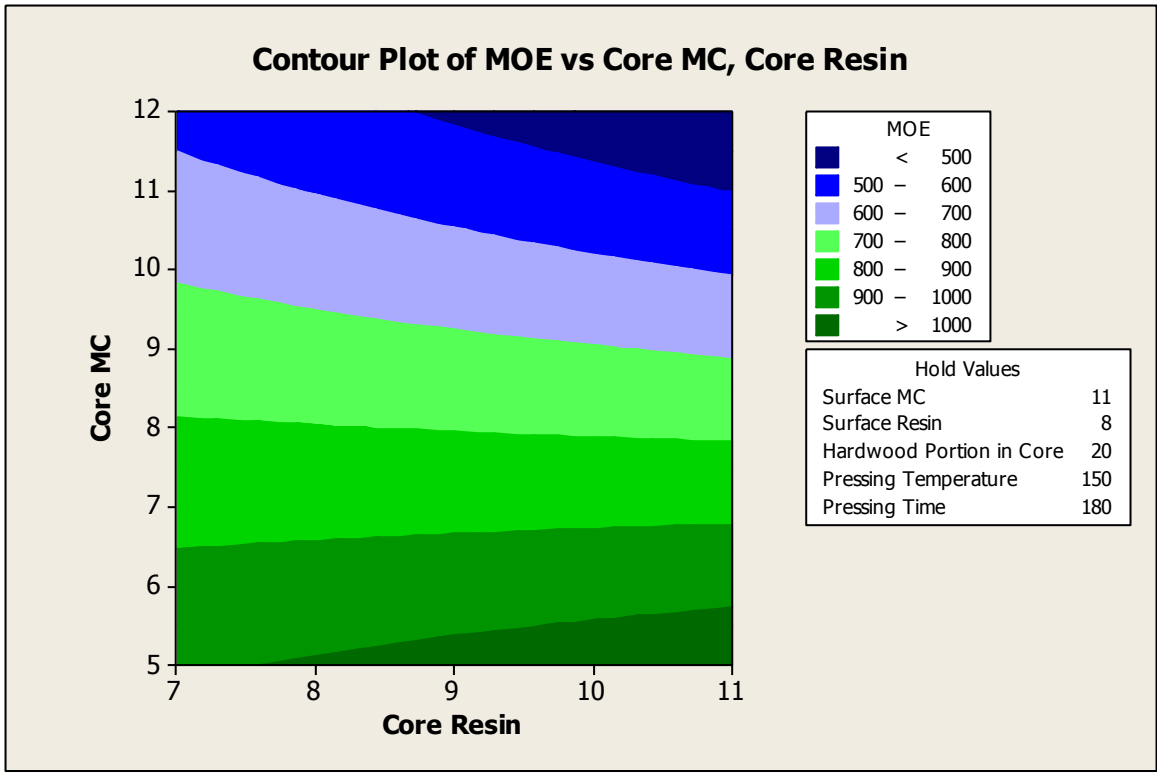


Figure 7.6 Contour plot of MOE with respect to core MC and core resin

The study of all these plots leads to a better understanding of significant factors that affect MOE, and the effect of each significant variable on MOE is revealed. Using contour plots against MOE, each variable at its ideal level is found. Better MOE can be achieved at non-uniform moisture distribution over the board thickness. Higher pressing temperature allows a shorter pressing time to achieve a better MOE. The hardwood portion in the core is insignificant to MOE, and higher resin content contributes to a better MOE.

7.6.2 Factors affecting Modulus of Rupture (MOR) of particleboard

Similar to MOE, the T and P values for each variable and important interactions with respect to MOR can be read in Table 7.4. The most significant variables and their interactions have $P < 0.05$ and have higher T values in the table. The higher the T value at $P < 0.05$, the higher the effect on MOR. Figure 7.7 demonstrates the normal probability plot for standardized effect for MOR. Significant parameters which control MOR are marked with red squares. The test statistical value for the effect that satisfies 95 % significant level is around (+ or -)

2.5, which is shown as a blue line in Figure 7.7. The significant variables which have a positive effect on MOR can be found to the right of the blue line, while the variables with negative effect on MOR lie to the left of the blue line.

Figure 7.8 shows the significant effects in a bar chart. Pressing temperature is the most significant variable affecting MOR, as it directly influences the resin curing process within the board. Pressing temperature must be controlled carefully as if the pressing temperature is higher or lower, an over-cured or under-cured resin will result in the boards. As the surface contributes most to MOR, adequate pressing temperature is essential to achieve optimal MOR.

The combination of core MC and core resin has the next most significant effect on MOR, negatively affecting the MOR of particleboard. This is discussed further in relation to the contour plot later in this section.

Surface MC and surface resin are the next two most significant factors on MOR. Similar to MOE, an appropriate surface MC assists heat transfer throughout the board. Resin plays an important role in binding all the particles together. Higher amounts of resin produce a strong and rigid composite, which also reduces the water absorption and thickness swell (Karr et al., 1999).

The combination of core MC and surface resin, core MC, the combination of core MC and hardwood portion in the core, and core resin are the remaining factors that positively affect MOR. On the other hand, pressing time, and the combination of core MC, surface MC and surface resin are factors that negatively affect MOR.

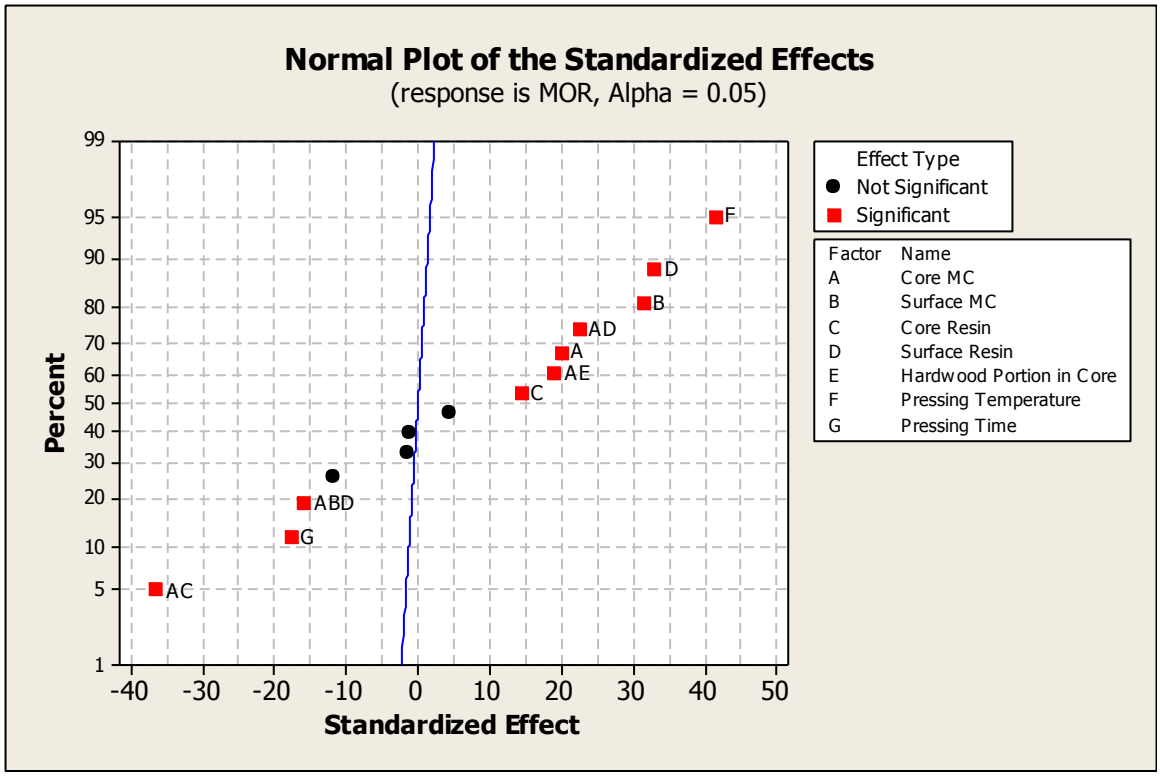


Figure 7.7 Normal probability plot for standardized effect for MOR

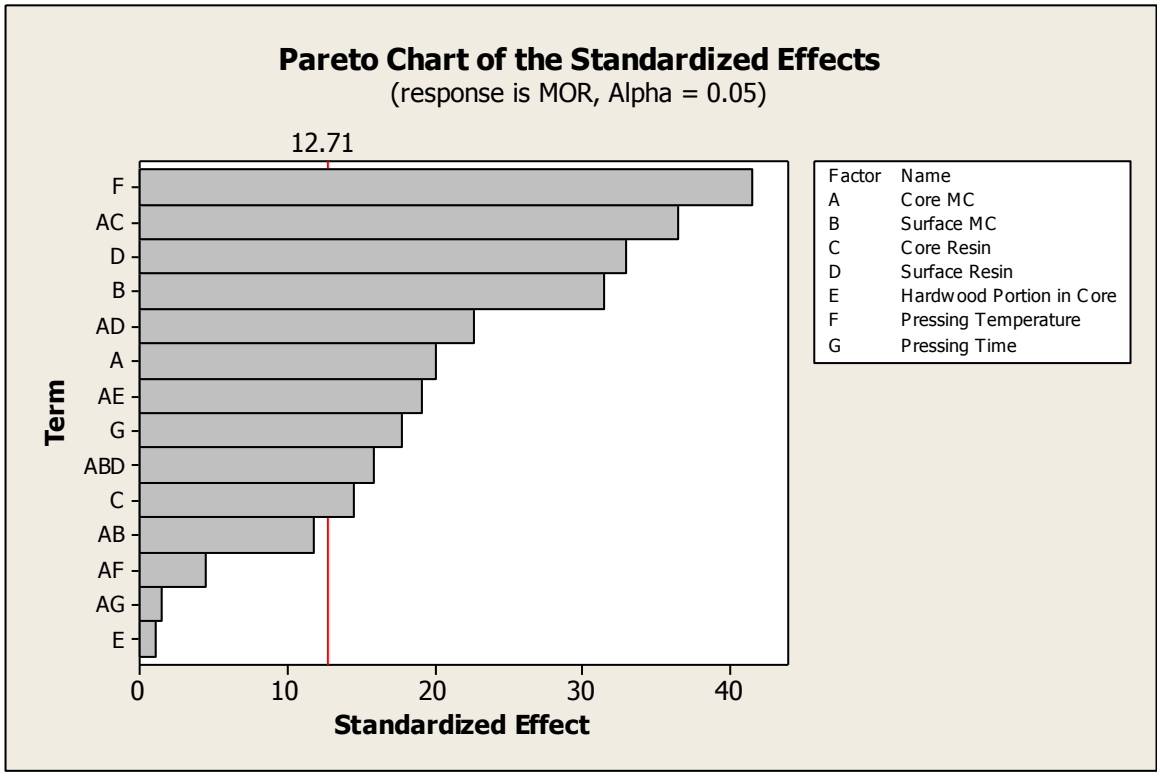


Figure 7.8 Pareto chart for standardized effect MOR

These data were then further analysed using contour plots to better understand the relationship between each significant variable with respect to MOR. In contour plots, two parameters are changed at a time, keeping other parameters constant at the middle of their range.

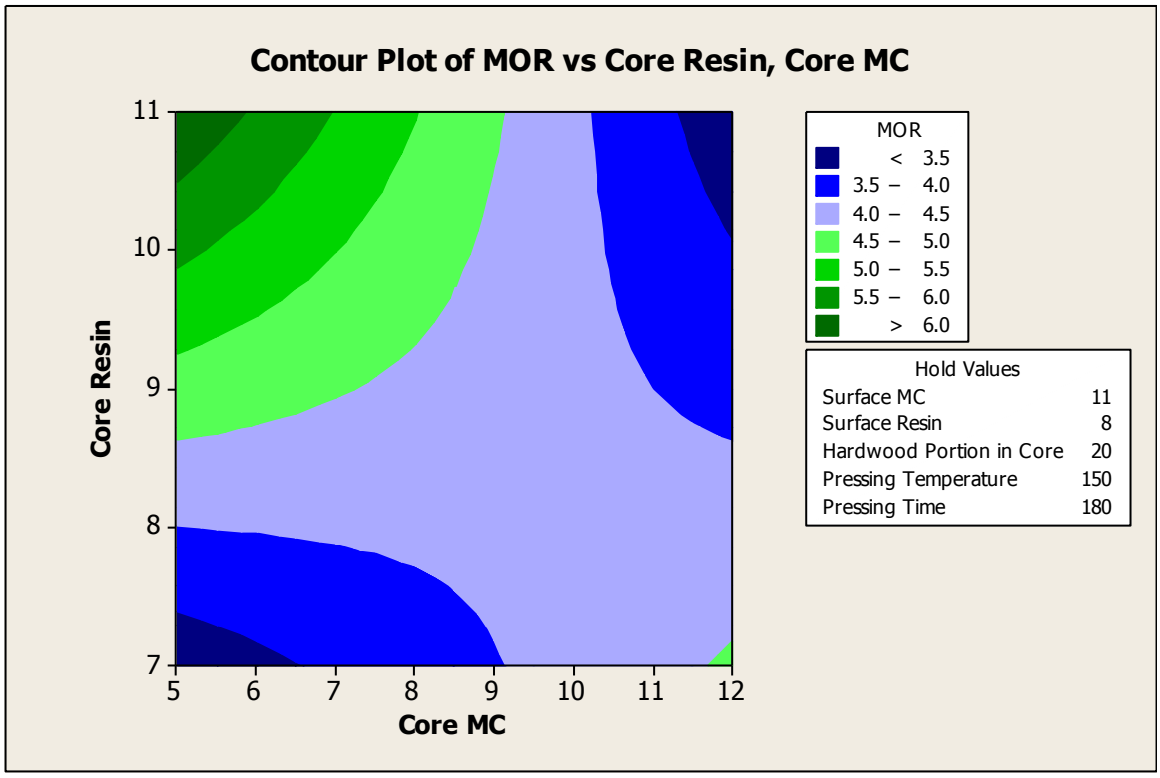


Figure 7.9 Contour plot of MOR with respect to core MC and core resin

The combination of core MC and core resin is the second most significant factor affecting MOR. The contour plot in Figure 7.9 clearly shows that the combination of core MC and core resin negatively affect the MOR of particleboard. As the core resin and core MC increase respectively, the MOR value decreases. However, by increasing the core resin gradually with the core MC maintained at the lower level of its range, the MOR value increases. This indicates that core MC must be maintained at the lower level of its range to prevent excess moisture being trapped in the core which may cause spring-back or thickness swelling.

Figure 7.10 shows the resultant MOR when moisture surface and resin surface are changed. Surface moisture is considered an important factor as it directly influences the heat transfer throughout the particleboard thickness for resin to cure in sufficient time. Similar to MOE, increasing the surface MC by keeping surface resin constant leads to a steady increase of MOR. However, by increasing the surface resin, while keeping the surface MC constant,

MOR does not change significantly. Researchers (Nirdosha et al., 2007, Myron, 1977) have found that lower moisture content can cause rapid drying of the glue spread due to the strong absorption of the water into the wood surface. High moisture content can lead to a high flow and enhanced penetration into the wood, causing starved glue lines.

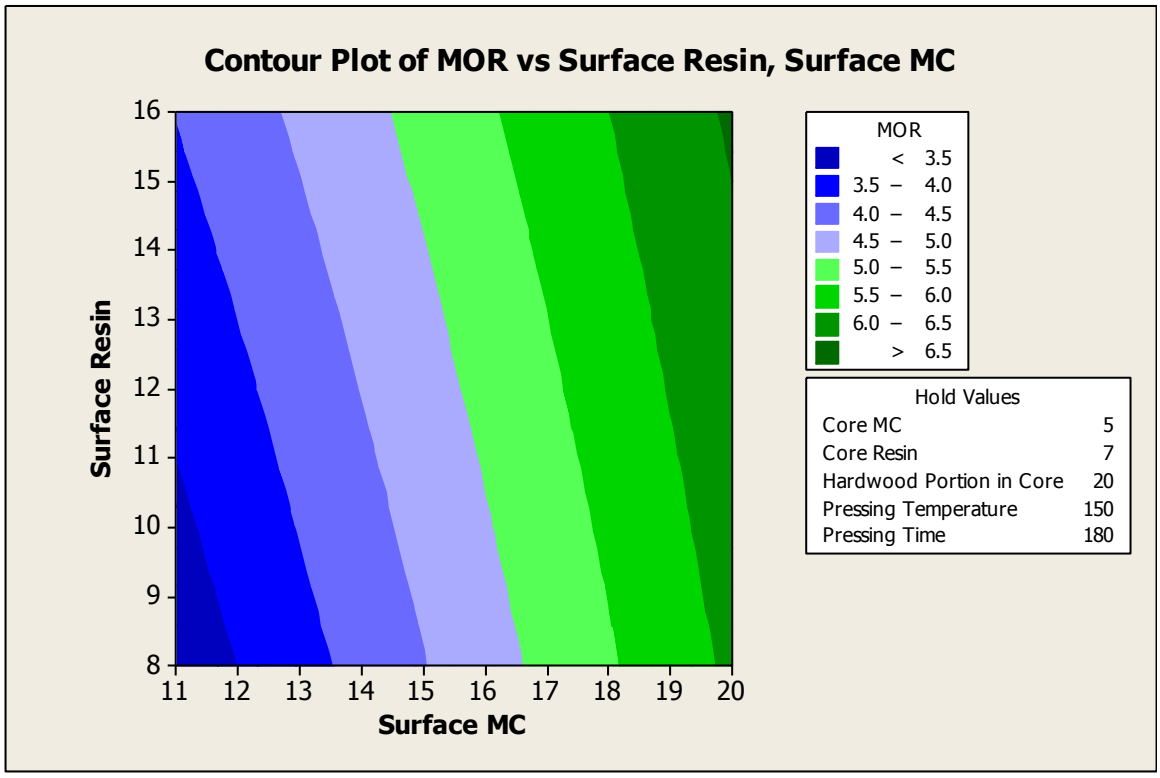


Figure 7.10 Contour plot of MOR with respect to surface MC and surface resin

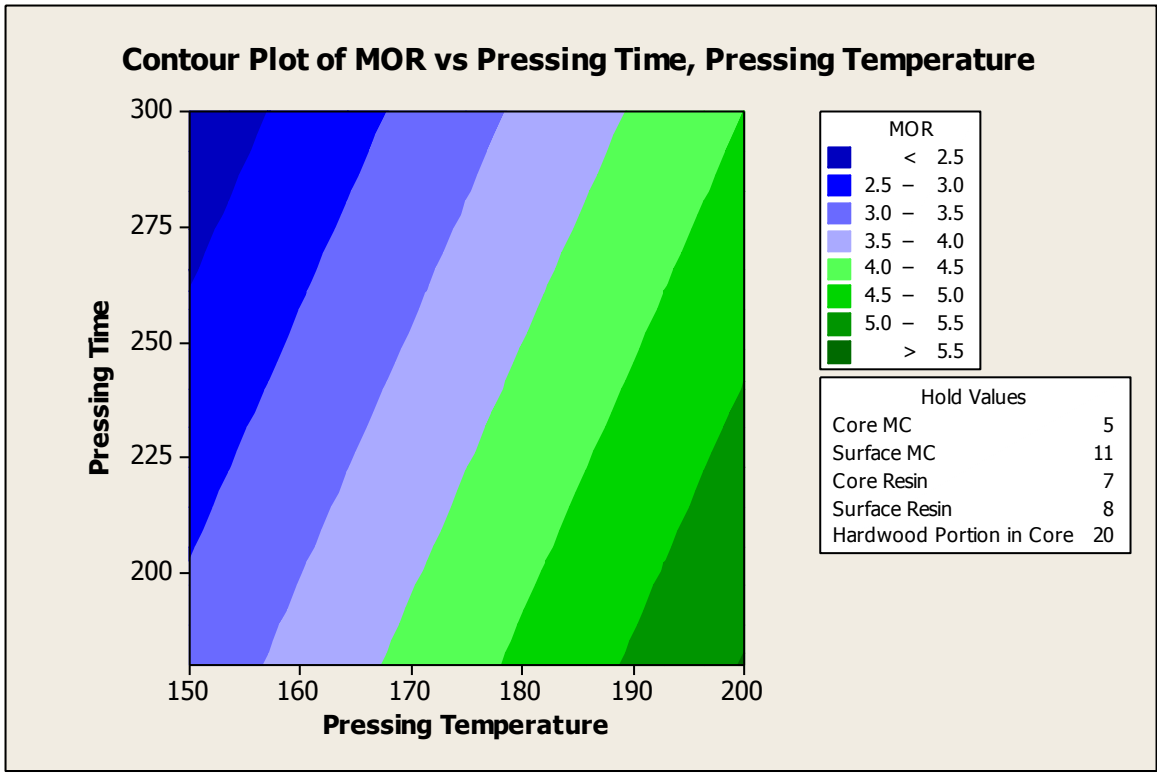


Figure 7.11 Contour plot of MOR with respect to Pressing temperature and Pressing time

Figure 7.11 exhibits the influence of interactions between press temperature and time on board properties. Similar to MOE, the MOR value is low at low pressing temperature and pressing time. The plot also shows that pressing temperature is more dominant as the MOR increases significantly by increasing the pressing temperature, while keeping the pressing time constant. Therefore, in order to obtain a higher MOR, the pressing temperature must be at a higher level.

The relationship between core MC and surface MC on MOR is clearly demonstrated in Figure 7.12. At low values of both core and surface MC, the MOR of particleboard is weak. Kollmann et al. (1975) reported that when moisture content is very low, the particles absorb more adhesive, which leads to a lack of adhesive on the particle surfaces. Excessive drying (hot press) may cause surface deactivation, which may result in poor adhesion. Similar to MOE, MOR increases when surface moisture is increased while keeping the core moisture at the lowest limit. This again confirms Hypothesis 2 that non-uniform moisture distribution over surface to core layers contributes to better board properties. A higher MOR value may be achieved with surface moisture at 18 % to 20 %, while keeping core moisture at 8.5 % to 12 %.

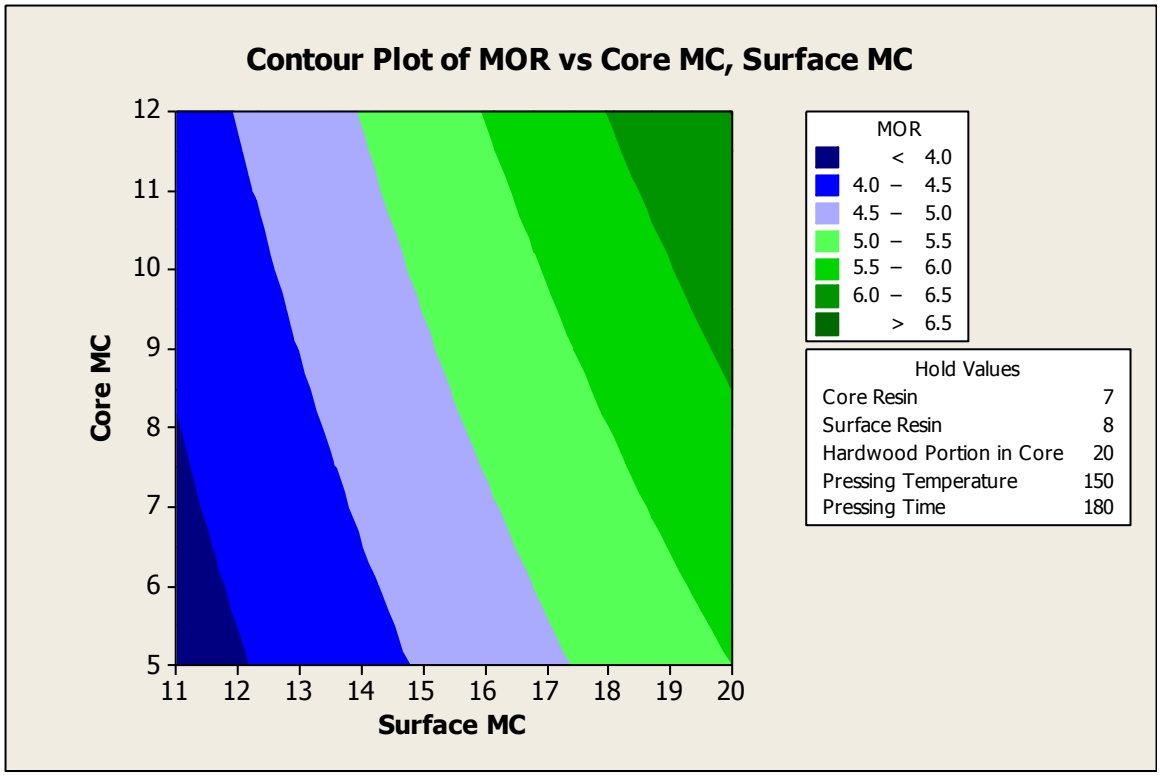


Figure 7.12 Contour plot of MOR with respect to core MC and surface MC

7.6.3 Factors affecting Internal Bond (IB) of particleboard

Internal bond strength (IB) is measured as the tensile strength perpendicular to the board's surface. When the tensile stress is applied to the surface, the particleboard normally fails in the middle where the density is low. This low density region is due to the low inter-particle contact and low consolidation.

Similar to MOE and MOR discussed earlier, the T and P values of each important effect with respect to IB are tabulated in Table 7.4. It is seen that the hardwood proportion in the core, the interaction of core MC and pressing time, and the interaction of core MC and hardwood portion in the core are the three significant effects with respect to IB (at $P < 005$).

Figure 7.13 indicates the normal probability plot of standardized effects for IB. Parameters with positive effects on IB are allocated on the positive side of the graph. It is obvious that only three significant factors have positive effects on IB.

The Pareto chart in Figure 7.14 indicates that the hardwood proportion in the core has the most significant effect on IB, followed by the interaction of core MC and pressing time, and

the interaction of core MC and hardwood proportion in the core. These significant variables are to be expected, as the IB strength of a particleboard is mainly dependent on the inter-particle bonding of the core.

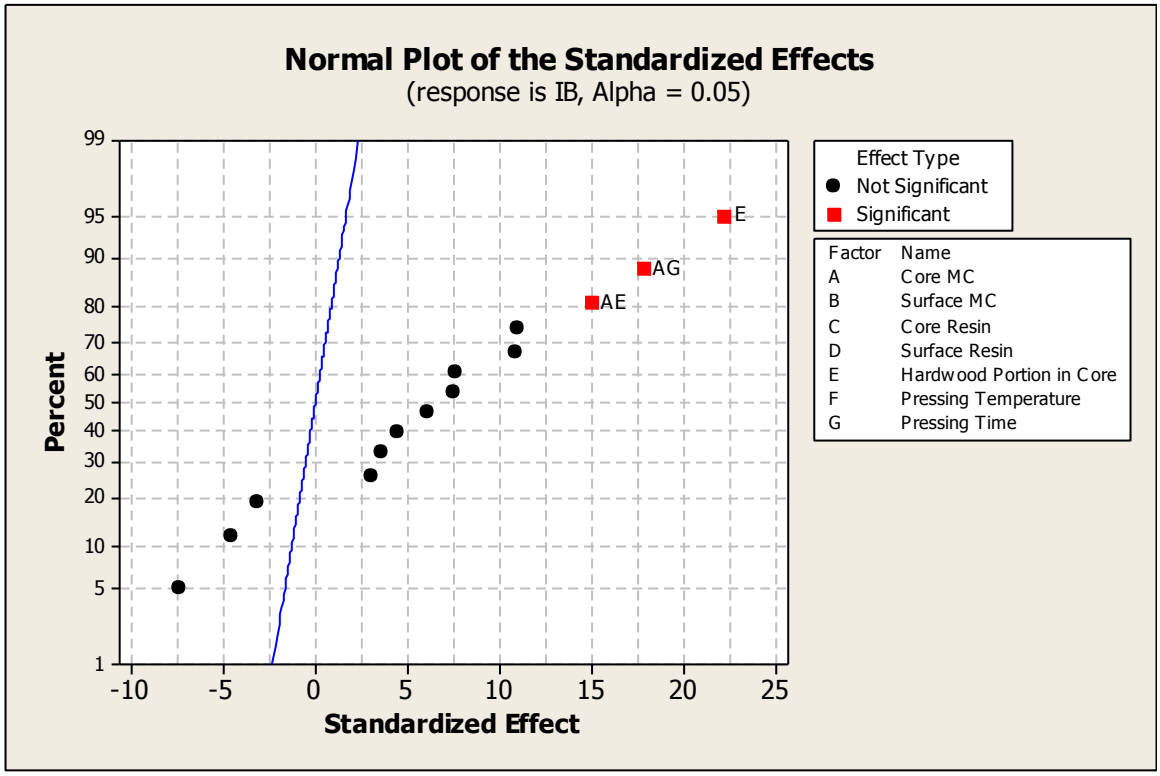


Figure 7.13 Normal probability plot of standardized effect for IB

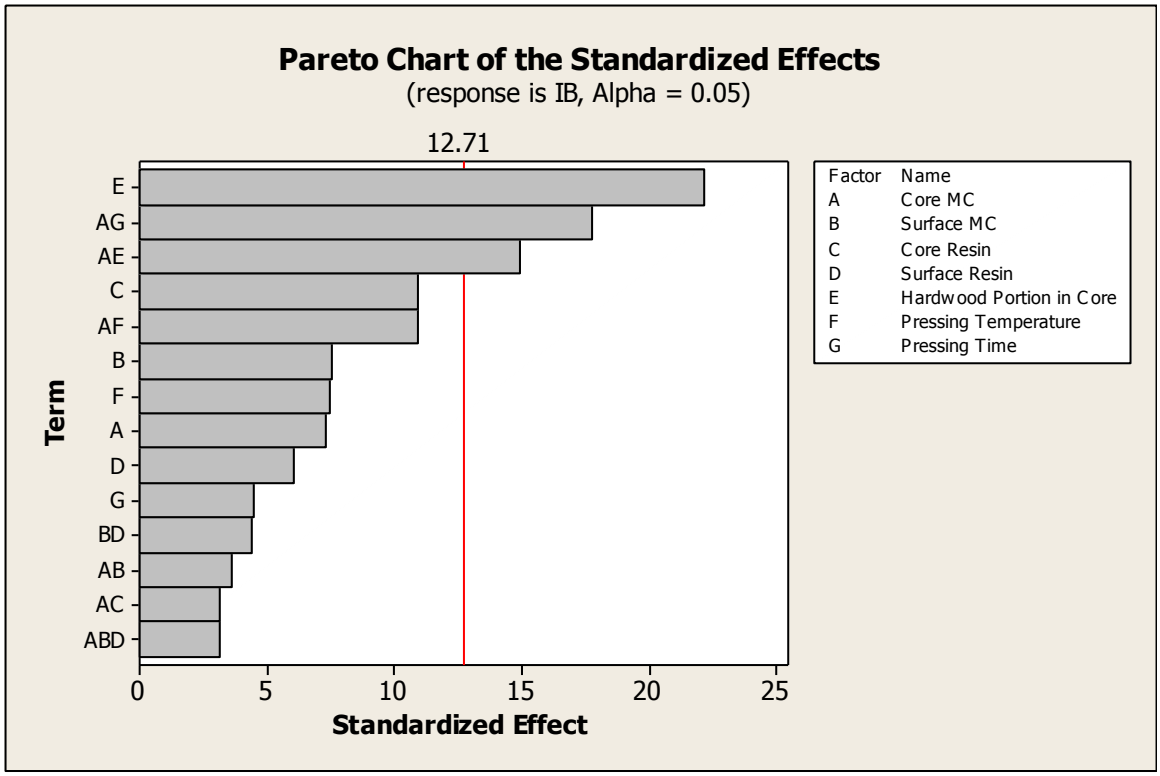


Figure 7.14 Pareto chart of standardized effect for IB

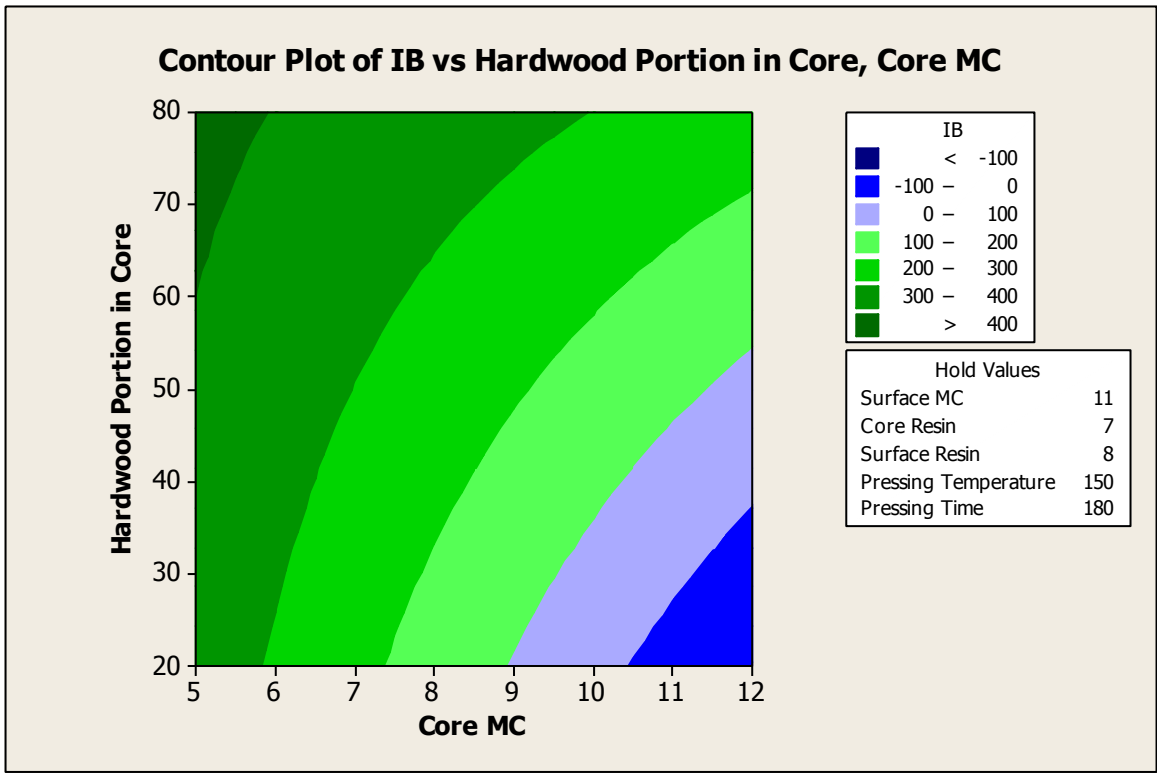


Figure 7.15 Contour plot of IB with respect to Core MC and Hardwood portion in core

Contour plots are used for a better understanding of the importance and relationship of significant variables. Figure 7.15 shows the relationship of hardwood proportion in the core

and core MC with respect to IB. As the moisture core increases by keeping the hardwood portion in the core constant, the IB decreases steadily. Therefore, it is preferable for the core MC to be maintained at its lower level for higher IB. Furthermore, a higher hardwood proportion in the core results in a better IB.

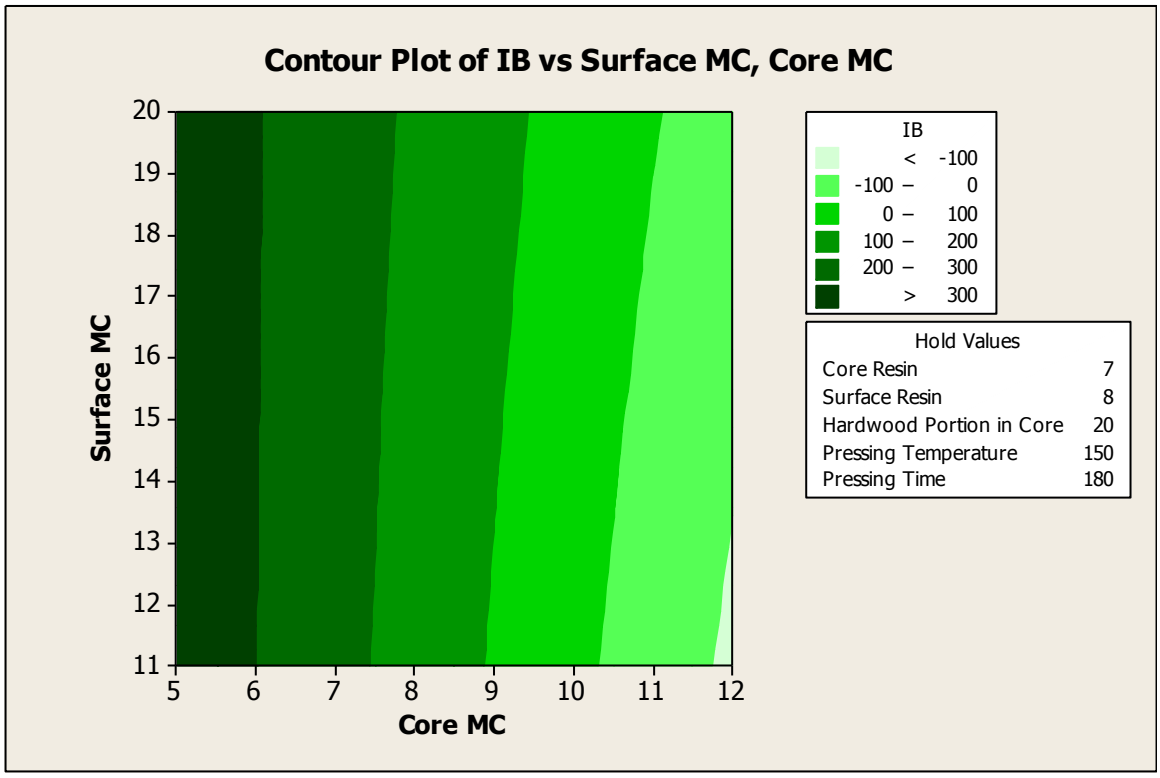


Figure 7.16 Contour plot of IB with respect to Surface MC and Core MC

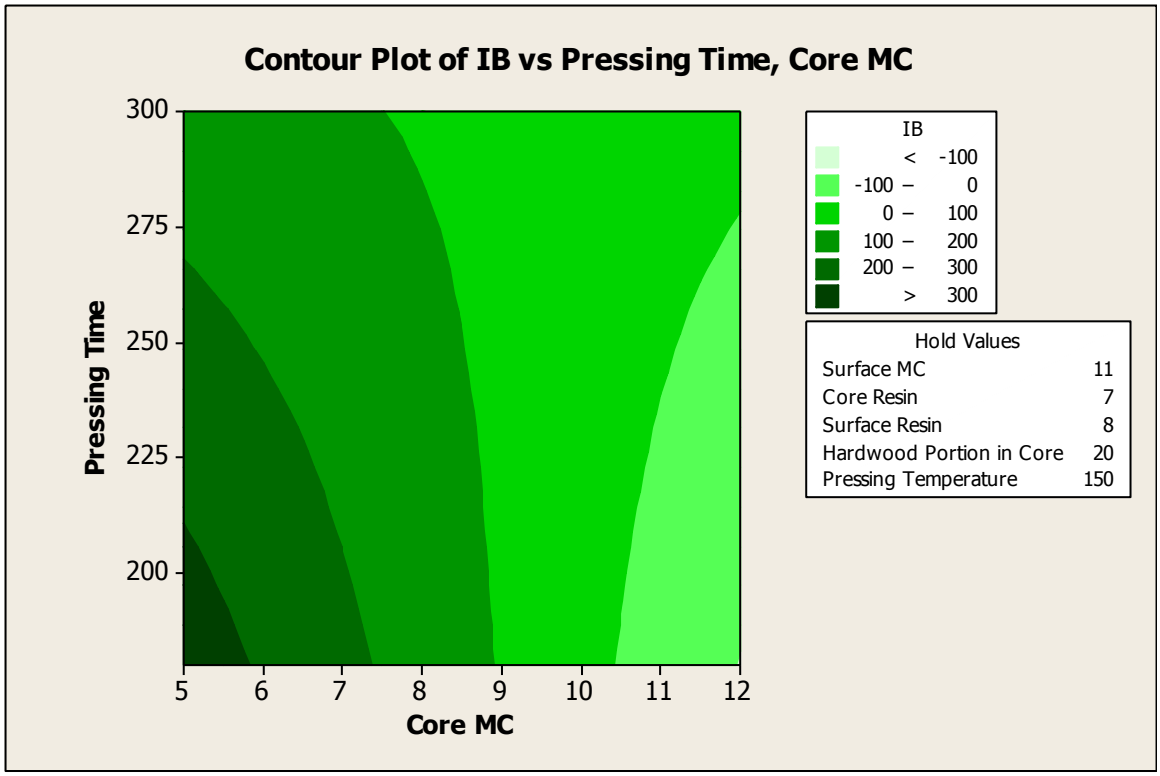


Figure 7.17 Contour plot of IB with respect to Pressing time and core MC

Once again, Figure 7.16 confirms that non-uniform moisture distribution over the surface and core layers improves particleboard performance. Core moisture core must be maintained at lower levels than surface moisture. It can be observed that as core moisture increases, the IB value decreases steadily.

The relationship of pressing time and core MC is clearly displayed in Figure 7.17. It is suggested to maintain core MC at a lower level to obtain a better IB strength. Adequate core moisture may lead to sufficient heat transfer without any excess moisture being trapped in the core during resin curing, which also shortens the pressing time. Figure 7.17 shows that lower core moisture gives shorter pressing time, which is efficient to produce an optimal IB strength. As the core moisture increases with pressing time, the IB strength is poor due to the excess moisture trapped in the core that would probably require a longer pressing time to remove.

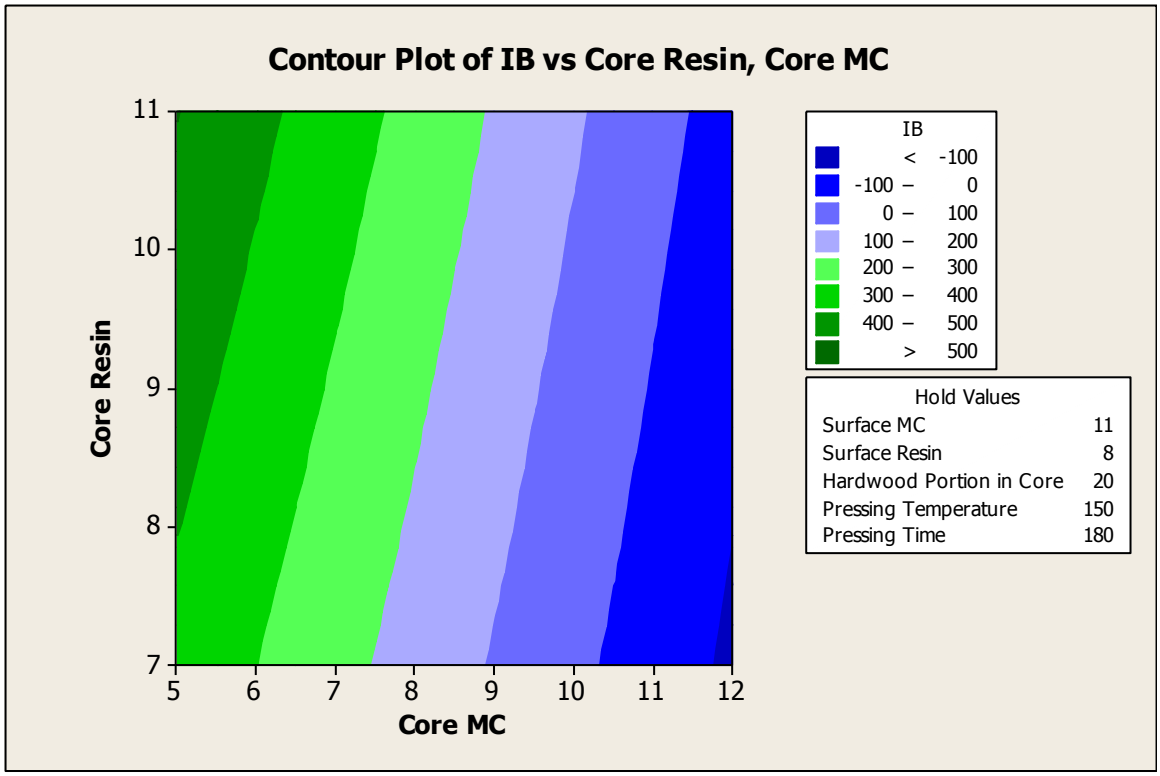


Figure 7.18 Contour plot of IB with respect to core resin and core MC

Figure 7.18 shows the behaviour of IB with respect to changes in moisture core and resin core. Increasing the moisture core reduces the IB, even if the resin core is kept constant. Therefore, core moisture should be maintained at lower levels. From Figure 7.18, it is clear that industrial grade IB can be found with core resin at 8 % or above if the core moisture is at its minimum.

7.6.4 Discussion of results in relation to hypotheses

As stated in Section 7.2.1, it was hypothesised that the use of hardwood residues in the surface layer would contribute to improved strength properties of particleboard. However, the results do not satisfy the requirements of the AS/NZS standards. By comparing the properties of the optimum boards produced from Series 1 and Series 2, as shown in Table 7.5, it can be observed that the MOE and IB values improve with higher board density.

Board	MOE (MPa)	MOR (MPa)	IB (kPa)	Density (kg/m^3)
Series 1 – Mixture of hardwood and softwood in surface and core layer	1730.457	14.977	329.376	728.742
Series 2 – Hardwood only in surface layers/mixture of hard/softwood in core layer	2178.931	10.464	780	732.746

Table 7.5 Properties of optimum board from Series 1 and Series 2

Cheng et al. (2006) investigated the medium-density fibreboard performance as affected by wood fibre acidity, bulk density and size distribution. The researchers found that if a longer hot-pressing time is used to cure UF resin mixed with lower pH fibres, the resin will be over-cured which will result in poor bond strength. Therefore, the properties of the hardwood may be one of the reasons why the results do not meet the standard requirements. The acidity of both softwood and hardwood residues was tested first as the acidity of the materials will affect the resin curing rate of the particleboard. The pH testing method was adopted from Stewart and Lehmann (1973). The results of the pH testing are shown in Table 7.6.

Sample	pH value
Distilled water	7.00
Hardwood mulch	6.24
Hardwood fine	5.13
Softwood mulch	6.74
Softwood fine	6.75

Table 7.6 pH test results of both hardwood and softwood residues

The pH test results show that softwood particles are slightly acidic or rather neutral as the pH is similar to that of distilled water. However, hardwood residues have an acidic nature with pH < 7.0. As discussed earlier in Chapter 2, particleboard produced with UF resin favours acidic conditions for faster curing, and hardener is commonly used in the softwood

particleboard industry to accelerate resin curing. Since only hardwood residues were applied in the surface layers for Series 2, this caused a difference in resin curing rate throughout the board. With only the acidic hardwood residues in surface layers, the resin within the surface layers would cure faster. The fast resin curing of surface layers may have caused over-curing of the surface layers over the press cycle. Due to the longer pressing time of the hardwood surface, the water in the wood cell antrum greatly reduces. As a result, wood fibres become fragile and easily damaged, and hydroxyl in wood becomes unstable with loss of water and the resultant reduction of free energy from the surface of wood (Zhao et al., 2007). This results in degradation of the wood particles and the breakage of some of the glue bonds. Cheng et al. (2006) found that if a longer hot-pressing time is used to cure UF resin mixed with lower pH fibres, the resin will be over-cured, which will result in poor bond strength. Consequently, the resulting properties of the particleboard will be poor.

Meanwhile, Hypothesis 2 was that non-uniform moisture distribution over the surface and core layers would give better board properties. This hypothesis has been verified by the contour plots of surface MC and core MC with respect to MOE, MOR and IB respectively. It has been shown that non-uniform moisture distribution at higher surface MC with lower core MC results in optimal properties. The higher amount of moisture in the surface than the core accelerates heat transfer to the core without unnecessarily lengthening the press cycle. However, several researchers have suggested that the surface MC should only be changed within a limited range. For example, Lehmann (1960) reported that an increase in surface moisture above 16.5 % reduced the MOR and IB.

Charles (2007) found that moisture content greatly influences the wetting, flow, penetration, and even cure of aqueous wood adhesives. Water has strong molecular attraction to wood, primarily through hydrogen bonding with hydroxyl groups of wood cellulosics. Therefore, the moisture content of adhesive must be controlled carefully. With lower wood moisture content, wood absorbs water from the adhesive so quickly that adhesive flow and penetration into the wood is drastically inhibited, even under high pressure and temperature. The wood may become so dry with continuous heating that it will resist wetting by the adhesive, because insufficient water remains bound to the wood to establish intermolecular attraction forces with the adhesive.

If the wood moisture content is excessive, less adhesive can be absorbed by the wood. This leads to excessive adhesive mobility, followed by over-penetration of the adhesive. During hot pressing, this excess moisture will cause high pressure to build internally as water boils, and on release of the platen pressure, the sudden release of internal pressure actually separates laminates along the bondlines, known as springback. Even if spring-back does not occur, the excess moisture will cause incomplete cross-linking of the adhesive with the wood particles. Hence, control of moisture content is particularly critical to bonding.

7.7 Summary

From the experimental study and an analysis of the factors affecting the mechanical properties of hardwood/softwood particleboard, it has been found that moisture is a critical variable that must be controlled carefully for better mat compaction, as moisture is the main medium that transports heat from the surface to the core of the board. Better board properties favour high moisture content at the surface with lower core moisture content. This non-uniform moisture distribution contributes to facilitating a shorter press cycle, as it accelerates heat transfer from the surfaces to the core for resin curing without any excess moisture being trapped.

The acidity of hardwood particles possibly leads to the rejection of the hypothesis that hardwood particles only in surface layers contribute to bending strength. The UF resin used in particleboard production was found to cure faster in acidic conditions. Therefore, with only hardwood particles in surface layers, the fast curing of surfaces results in over-curing of the layers which degrades the glue bonding between particles.

Pressing time and pressing temperature are inversely proportional with respect to the mechanical properties of particleboard. At higher pressing temperatures, the pressing time can be adjusted to be relatively shorter, as high temperature would promote a faster heat transfer and increase the curing rate. Particleboard produced at high temperature and long pressing time would have some resin over-curing, which will lower the strength properties of particleboard.

The results also indicate that high proportions of hardwood in the core result in a particleboard with a better tensile strength. This can be explained by the acidity of hardwood

particles. A higher proportion of hardwood in the core facilitates faster curing in the core layer. Since the core layer may take longer to reach a sufficient resin curing temperature, the acidity of hardwood helps the core resin to cure in time without lengthening the pressing period.

Resin is the main ingredient that creates permanent inter-particle bonds in the particleboard. Therefore, adequate resin content is vital for the mat to consolidate efficiently within the pressing time. Core resin at higher levels result in better IB strength.

Chapter 8

IDENTIFYING APPROPRIATE CONFIGURATIONS OF HARD/SOFTWOOD PARTICLES IN CORE AND SURFACES: SERIES 3

8.1 Introduction

The analysis of the results of the Series 2 experiments in the previous chapter has demonstrated that the acidity of hardwood particles is one of the critical factors affecting the quality of particleboard. The acidity of particles directly affects the resin curing of particleboard. With only hardwood residues in surface layers, a varying resin curing rate was found throughout the board. The surface layers, which are directly in contact with the hot platen, are expected to cure faster than the core layer. The application of acidic hardwood particles in the surface layers accelerates the resin curing of the surface layers. During hot pressing, the surface layers were thought to be over-cured and the glue-bonds destroyed.

In addition, many researchers have indicated that wood acidity has direct effects on the gel time and curing behaviours of resins (Albritton and Short 1979; Guo et al. 1998; Johns and Niazi 1980; Peng and Li 1983; Park et al. 2001; Pizzi et al. 1994; Pizzi and Panamgama 1995; Slay et al. 1980; Van Niekerk and Pizzi 1994; Xing et al. 2004b, 2005; Zanetti and Pizzi 2003). Cheng et al. (2006) found that the lower the pH of the particles, the shorter the hot-pressing period. However, if a longer hot-pressing time is used to cure UF resin mixed with lower pH fibres, the resin will be over-cured which will result in poor bond strength.

Therefore, it was decided that the acidity of the wood should be monitored to allow adjustments in the adhesive system to maintain the same rate of polymerisation throughout the board. The adhesive is pH-sensitive and excessive fluctuations in the wood pH may retard or speed the polymerisation process.

Since the pH variation among softwood and hardwood would alter the board properties, it is essential to examine the configuration of hardwood/softwood particles within the particleboard.

8.2 Objectives of Series 3 experimental study

According to the findings of previous researchers (Myron, 1977), the hot pressing period can be reduced when the furnish pH is low. This indicates that acidic particles are more effective in producing particleboard with a short pressing time. However, there will be a variation in the degree of resin curing throughout each layer of particleboard. The surface layers will cure better than the core layer, and the surface layers will possibly be over-cured if the pressing time is not controlled appropriately.

From Table 7.6, it was found that hardwood particles have a lower pH than softwood particles. The acidity of hardwood particles is expected to produce a better board. However, the results in Series 2 did not confirm this expectation. This uncertainty might be due to the over-curing of the hardwood surface layers which destroyed the glue-bonds.

Therefore, the configuration of hardwood/softwood particles in core and surfaces will be investigated in this section in order to find:

- The degree of curing as affected by the configuration of hardwood/softwood in the core and surface layers
- The properties of particleboard as affected by the configuration of hardwood/softwood in the core and surface layers

8.3 Experimental design

Based on the analysis and discussion of the work presented in previous chapters, six different configurations of hardwood/softwood particles in core and surfaces were identified for investigation. Table 8.1 shows the selected hardwood/softwood configurations in core and surfaces of particleboard.

Board no.	**Configuration**
1	Hardwood only in both surface and core layers **(H)**
2	Softwood only in both surface and core layers **(S)**
3	Hardwood only in surface/softwood only in core **(H-S)**
4	Mixture of hardwood/softwood in surface/hardwood only in core **(HS-H)**
5	Softwood only in surface/hardwood only in core **(S-H)**
6	Softwood only in surface/mixture of hardwood/softwood in core **(S-HS)**

Table 8.1 Hardwood/softwood configurations in core and surfaces

Mixtures of hardwood/softwood were decided at the ratio of 60 % hardwood/40 % softwood. Processing parameters were maintained constant as per the findings presented in Chapter 7. Table 8.2 illustrates the processing parameters that were chosen to be constant. It was decided to apply non-uniform moisture distribution with higher moisture at the surface (20 %) and lower moisture in core layers (5 %). Resin surface and core were chosen to be at higher levels in order to obtain better bonding between particles. 240 seconds of pressing time was selected with pressing temperature at 200^{o}C.

Variables	Units
Moisture Surface	20% of dry wt of the board
Moisture Core	5% of dry wt of the board
Resin Surface	16% of resin load as a % of dry wt of the board
Resin Core	11% of resin load as a % of dry wt of the board
Pressing Temperature	200^{o}C

Pressing Time	240 seconds

Table 8.2 Process parameters constant for particleboard production

Although maintaining these variables at constant values did not allow the identification of ideal board properties, this subsidiary series of experiments assisted in identifying the best configuration of softwood and hardwood for optimum board properties.

8.4 Materials and methodology

The same processing procedures were utilised as described in Section 6.3.2. Mixtures of hardwood and softwood particles were applied only in particleboard with the configuration of S-HS and HS-H.

As described in Section 6.3.3, the finished three-layer boards were then tested in accordance with AS/NZS. Samples of 50 mm x 50 mm were prepared from each board for IB testing, while MOR and MOE were tested with samples of 100 mm x 300 mm.

The only change to the process presented in Chapter 6 was the application of AS/NZS 4266.21:2004 for the testing of the degree of curing of each particular board. The following procedure was adopted for the resin curing test:

- Test pieces of 100 mm x 100 mm were prepared and conditioned
- Sulphuric acid at 0.2 N and Rhodamine red dye were used for the testing
- Test pieces were cleaned if necessary
- A watch glass was inverted onto the surface of the test piece. About 1mL of acid was dispensed containing a small amount of the dye under the watch glass onto the test piece surface. The edge of the watch glass was then lowered to prevent acid escaping.
- Timing was then commenced for 2 hour (+ or – 5 min).
- The watch glass was then removed and the acid washed off acid with cold water.
- The surface was then wiped dry and the specimen set aside for visual grading.

The degree of curing of each board was graded according to the following ratings:

(i) Rating 1 – faint marking, no surface rise or loss of gloss (over-cure)

(ii) Rating 2 – slight marking, slight surface rise and loss of gloss

(iii) Rating 3 – moderate marking, moderate surface rise but still gloss in places

(iv) Rating 4 – definite marking, definite surface rise and surface gloss has vanished

(v) Rating 5 – very definite marking, very definite surface rise (under-cure)

Calipers were used to measure the surface rise by measuring the displacement of rise area.

8.5 Results and discussion

The results of the particleboards produced in different configurations are tabulated in Table 8.3. MOE, MOR, IB and density are the four major outcomes considered.

Board	**MOE (MPa)**	**MOR (MPa)**	**IB (kPa)**	**Density (kg/m^3)**
1 - **(H)**	1840.951	9.678	164.50	661.95
2 – **(S)**	2635.505	17.539	553.248	728.63
3 – **(H-S)**	323.7502	3.437	37.876	833.26
4 – **(HS-H)**	944.4404	6.260	138.376	686.93
5 – **(S-H)**	3237.201	16.903	876.876	740.56
6 – **(S-HS)**	2510.907	11.657	671.00	742.82

Table 8.3 Results of particleboards with different hardwood/softwood configurations

The degree of curing of each board was tested as discussed earlier. Table 8.4 shows the results of the curing tests.

Configuration	Observation	Indication
Hardwood only **(H)**	Loss in gloss on surface No surface rise	Over-cure
Softwood only **(S)**	Gloss in place on surface Surface rise: 15 to 15.2 mm	Perfect cure
Hardwood surface/softwood core **(H-S)**	Loss in gloss on surface No surface rise	Over-cure
Mixture soft/hardwood surface/hardwood core **(HS-H)**	Loss in gloss on surface Surface rise: 17 to 19 mm	Under-cure
Softwood surface/hardwood core **(S-H)**	Gloss in place on surface Surface rise: 15.55 to 16 mm	Perfect cure
Softwood surface/mixture of hard/softwood in core **(S-HS)**	Gloss in place on surface Surface rise: 15.2 to 15.5 mm	Perfect cure

Table 8.4 Curing level of particleboards with different hard/softwood configurations

Only the surface layer was considered for this curing test as the testing was only applied to the surface layer and observations were only taken on the surface. Table 8.4 indicates the observations on the sample surfaces.

8.5.1 The properties of particleboard as affected by the configuration of hardwood/softwood in core and surface layers

The optimal hardwood/softwood configurations in the surfaces and core can be seen in Table 8.3. Particleboards with softwood only in the surfaces result in better board properties. Boards with the configuration of **S – H** have optimal properties at 3237.2 MPa (MOE), 16.9 MPa (MOR), and 876.87 kPa (IB). These mechanical properties easily fulfill the requirement of Australian Standard AS/NZS 4266.

In comparing the results of hardwood and softwood particleboard, softwood particleboard obtains better board properties. This result shows the effect of wood species on board properties. Several researchers stated that board properties may differ for different mat formations, even with the same wood particle size distribution. For instance, Larmore (1959) compared the difference of properties between board made from aspen furnish (specific gravity 0.37) and yellow birch (specific gravity 0.65) respectively. It was found that the modulus of rupture (MOR) for aspen furnish particleboard is higher than yellow birch board. This finding clearly illustrates that particles with lower specific gravity give better board properties. Therefore, softwood residues at lower specific gravity than hardwood residues obtain better properties, as seen in Table 8.3.

Fabricio et al. (2005) found that a lower density wood provides a high density compaction rate and, therefore, more contact surface between the particles than high density wood. This leads to a more uniform product with a greater capacity to transmit loads between the particles, resulting in higher flexural and internal bonding properties in particleboards made of low density wood. Therefore, particleboard from lower density softwood has better properties.

The effect of particle size may also explain the difference in properties between hardwood and softwood particleboard. Osarenmwnda et al. (2007) investigated the effect of particle size on some properties of rice husk particleboard. The researchers found that as the particle size increased, the particleboard's mechanical and physical properties decreased. Therefore, it can be concluded that the smaller the particle size, the better the properties of the particleboard, as smaller particles result in better alignment, minimizing the inter-particle voids. As seen in Figure 5.3 and Figure 5.4, softwood particles are relatively smaller than hardwood particles and hence softwood particleboard has undoubtedly better properties.

Gertjejansen and Haygreen (1973) compared particleboards made from 1.5 inch long wafers and 0.5 inch flakes, and found that the MOR reduced approximately 9 % when wafers were used instead of flakes. This again confirms that smaller particles (0.5 inch flakes) give better properties of particleboard.

The results indicate that board with S – H configuration has optimal properties. Research has shown that large particles at the board surface normally do not produce as smooth a surface as board with smaller particles. One widely used compromise is a three-layer construction in

which small particles, for smoothness, are used for particleboard surfaces and larger particles, with less adhesive, are used in the core (Heebink et al., 1964).

The different acidities of hardwood and softwood can also explain the optimal properties of board with the S – H configuration. As discussed earlier, hardwood particles have lower pH, which may accelerate the curing of UF resin as UF resin favours acidic curing conditions. Libin et al. (2010) investigated the effect of the pH value of wood on the curing reaction of low toxic UF resin. Differential scanning calorimetry (DSC) was used for this study. Three wood species (poplar, birch, larch) with different pH values were used, and the results showed that the effects of different wood flour and its pH value on the curing reaction of UF resin were not the same. Along with the increase of the pH, the peak temperature and the onset temperature were raised. Therefore, hardwood and softwood particles may have different curing temperatures when reacting with UF resin.

During hot pressing, surface layers normally cure first as these surfaces directly contact the hot platen; the core layer will then cure with the assistance of heat transfer through moisture. Due to the variation in the resin polymerization rate throughout the board layers, the configuration of hardwood and softwood throughout the board must be monitored carefully. Since the core layer takes longer to reach the appropriate curing temperature, hardwood is more suitable to be allocated to the core layer due to its acidity with lower curing temperature and shorter curing time, while softwood, which needs more time to cure, is better in the fast heating surfaces.

However, board density was higher than expected in all the boards. Myron (1977) stated that the final average density of particleboard is not only dependent upon the amount of wood material in the mat but also upon processing conditions prior to the pressing operation, specifically, furnish species, preparation and drying, adhesive and moisture distribution. Therefore, further investigation is essential to lower the density of the board without affecting its properties.

8.5.2 The degree of curing as affected by the configuration of hardwood/softwood in core and surface layers

Table 8.4 clearly shows the curing level of each particleboard with different hardwood/softwood configurations. Although the curing test is only applicable to the surface layer, it can sufficiently differentiate the curing levels of hardwood and softwood.

Many studies have shown that UF resin curing is affected by wood species, pH, the buffer capacity of wood, and the soluble-acid and bound-acid contents. For example, Zheng et al. (2007) used differential scanning calorimetry (DSC) to characterize UF curing. With dynamic scanning of UF resin with a single-heating-rate method, DSC analysis can provide ample information related to resin curing, such as total reaction heat, onset temperature, peak temperature and activation energy. The effect of wood extracts on UF curing was investigated in this study. Wood extracts were collected from 16 wood species with different chemical properties, and the interactions of each wood extract and UF resin were measured by dissolving the extract in the resin before DSC scanning. When UF curing is evaluated by DSC, the peak temperature is treated as an indicator of the UF resin curing rate. It was found that UF resin in the presence of higher pH species needs to be heated to a higher reaction temperature. Species with lower pH are favourable for UF curing because acidic conditions are preferable for the curing reaction of UF resin.

The findings of Zheng et al. (2007) explain the difference of curing levels between hardwood and softwood. Since hardwood is more acidic than softwood, as shown in Table 7.6, hardwood has a higher curing rate or lower peak temperature. However, the higher curing rate of hardwood is not always an advantage for resin curing. As discussed in Section 7.6.4, if a longer hot-pressing time is used to cure UF resin mixed with lower pH fibres, the resin will be over-cured, which will result in poor bond strength. Therefore, it can be seen in Table 8.4 that the particleboards with hardwood only surfaces were found to be over-cured. Consequently, the resulting properties of the particleboards were correspondingly poor.

However, it is not clear that hardwood always has a higher curing rate than softwood. Vast differences in the nature and amount of various chemical components within wood extracts can be found even within a single species, depending on the age and growth site of the trees

sampled and the sampling location within the log. Therefore, it is essential to understand the nature of wood materials for particleboard production.

8.6 Summary

This subsidiary series of experiments utilized previously optimized processing parameters in making six boards with hardwood/softwood configuration as the single variable. The analysis confirmed the following results:

- Particleboard made with hardwood in the surface has poor board properties compared to boards with softwood only surfaces.
- The above observation partially confirms the hypothesis that hardwood in the surface leads to over-curing of particleboard.
- Further analysis is required to understand the curing of resin in hardwood/softwood particleboard so that an optimized board can be produced.
- Optimum board should have softwood only in the surface and hardwood only in the core

Chapter 9

PARTICLEBOARD PRODUCTION USING THE OPTIMISED S – H CONFIGURATION: SERIES 4

9.1 Introduction

The results in Chapter 8 have indicated that the S – H configuration, with softwood only in the surface and hardwood only in the core, is the best configuration for particleboard made from mixtures of softwood and hardwood. It was observed that with the S – H configuration, perfect curing happens within the board and hence better properties can be obtained. Since the core layer takes longer to reach the appropriate curing temperature, hardwood is a more suitable raw material in the core due to its acidity, with lower curing temperature and shorter curing time; while softwood, which needs more time to cure, is better in the fast heating surfaces.

However, the board density of the optimized board was still slightly higher than the AS/NZS standard. Therefore, a further investigation was carried out to lower the board density without affecting its properties. This chapter presents the final optimization process.

Some alterations were made to the selected variables for further investigation. Screening testing was then carried out to obtain any variation of or improvement in board density. Process modelling was then used to develop a model with respect to each response for board optimization.

9.2 Process modelling for board optimisation

Process modelling using experimental design is another important tool that can be used to develop a new product, to formulate a new process or to improve an existing product or process. It is an empirical method of developing a process model based on observed data from the process considering the response surface. It is also a well-recognized method in chemical and polymer science, and pharmaceutical and drug development, as it is an efficient and economical tool to develop almost any product. In addition, the model can be used to optimise particleboard properties and the recipe to produce particleboard with S – H configuration with the expected properties. The developed models were validated using further experiments.

9.2.1 Composite process models

Composite process models are empirical models developed using multiple regression analysis. Multiple regressions use a collection of statistical techniques for model building. The method is suitable for the exploration of response surfaces and to develop second order polynomial models, thus helping optimization of the process by using a smaller number of experimental runs (Myers and Montgomery 2002). The model generated contains quadratic terms with two-factor interaction effects of individual terms. The models are of the following form:

$$Y = a_0 + a_1x_1 + a_2x_2 + a_3x_3 + \ldots + a_8x_1x_2 + a_9x_1x_3 + \ldots a_n{x_1}^2 \ldots + a_m{x^2}_7 + E \qquad \text{Equation 9.1}$$

where,

Y is the selected response,

$a_0 \ldots a_m$ are the regression coefficients,

$x_1 \ldots x_7$ are the factors being studied and

E is the error term

MINITAB was used to calculate the regression coefficients for each response incorporating design of experimental theories. Once models were developed, the reliability of the models was estimated statistically before experimental testing.

9.3 Screening test

Based on the results and discussion presented in Chapter 8, the S – H configuration was identified as the best configuration for particleboard production using mixtures of hardwood and softwood residues. However, the density obtained was still slightly higher than the AS/NZS standard. In order to improve or lower the board density without affecting its properties, a screening test was developed, as presented in Table 9.1.

In three of the previous experiments, it was found that most of the materials (wood residues, resin) were utilised without any waste. Therefore, in order to lower board density, it is considered to eliminate the 20 % wastage that was applied on each of the previous series of experiments. After referring to previous results and further discussion with particleboard specialist, Dr Nirdosha Gamage, pressing time and pressing temperature were selected to be kept at 180 sec and 200^{o}C respectively. Only four variables at two levels were identified for screening tests, as seen in Table 9.1. Moisture surface (A), moisture core (B), surface resin content (C), core resin content (D) are the identified variables.

Variables	**Low Value**	**High Value**	**Units**
Moisture surface (A)	9	18	% of dry wt of the board
Moisture core (B)	7	12	% of dry wt of the board
Surface Resin Content (C)	8	16	Resin load as a % of dry wt of the board
Core Resin Content (D)	7	11	Resin load as a % of dry wt of the board

Table 9.1 Variables used for screening test

Moisture distribution was set at non-uniform over surface to core layers. Following verification in Chapter 8, only softwood was used in surface layers, while hardwood only was used in the core.

9.4 Experimental design

A screening experimental series was conducted to lower the density of board with S – H configuration without affecting properties. As Table 9.1 indicates, four variables at two levels

were identified to obtain a screening experimental series at ½ fractional (2^{4-1}) factorial design. Eight different experimental boards were produced in the RMIT laboratory. The different treatment combinations with respect to this 2^{4-1} design are tabulated in Table 9.2.

Board no.	MC Surface	MC Core	Resin Surface	Resin Core
1	9	12	16	7
2	9	12	8	11
3	18	12	16	11
4	9	7	16	11
5	18	12	8	7
6	18	7	16	7
7	9	7	8	7
8	18	7	8	11

Table 9.2 Different treatment combinations with respect to 2^{4-1} design

It was expected to obtain a target board density constant at 680 kg/m^3 with 15.2 mm thickness.

9.5 Materials and methodology

As for three-layer particleboard production, which was discussed earlier in previous chapters, the same materials were used to prepare particleboard for this investigation. The main difference was the content in both surface and core layers. It was decided to have softwood only in surface layers, while the core layer would contain only hardwood residues.

After discussion and consideration, the 20 % wastage applied in previous series was eliminated from every board in this series in order to accurately estimate the board density. Both cold and hot pressing times were kept constant at 180 sec. The measurement of the pressing time was commenced as soon as the top platen of the press touched the particleboard mat. The hot press temperature remained at 200^{o}C.

The laboratory procedure for the particleboard manufacturing was clearly explained in Chapter 6.3.2. All the completed boards were then sampled, conditioned and tested for their physical and mechanical properties.

As explained in Section 6.3.3, the finished three-layer boards were then tested in accordance with Australian Standard of testing AS/NZS. Samples of 50 mm x 50 mm were prepared from each board for IB testing, while MOR and MOE were tested using samples of 100 mm x 300 mm. The mean density of each board was also measured.

9.6 Results and discussion

Table 9.3 shows the eight different recipes given by experimental design for the manufacture of particleboards. Moisture content and resin content were calculated with respect to the oven dried wood weight and these percentage values are tabulated in Table 9.3. The averages of the test results are also tabulated in Table 9.3. MOE and MOR are measured in megapascals (MPa) while IB is measured in kilopascals (kPa). Mean density was also measured and the results are presented in Table 9.3.

Board no.	**MC Surface**	**MC Core**	**Resin Surface**	**Resin Core**	**MOE**	**MOR**	**IB**	**Density**
1	9	12	16	7	2294.39	13.5593	606.252	733.116
2	9	12	8	11	2294.39	13.0571	631.252	726.677
3	18	12	16	11	2824.26	16.4846	906.252	696.385
4	9	7	16	11	2581.2	12.8061	643.752	670.949
5	18	12	8	7	2064.95	9.0395	518.752	610.047
6	18	7	16	7	2346.55	12.8061	731.252	628.625
7	9	7	8	7	1923.24	9.5417	393.000	695.543
8	18	7	8	11	2067.20	9.2907	443.752	738.392

Table 9.3 Experimental variables and results

The test results show an obvious improvement in board properties. A comparison with Table 6.2 shows that almost every single board obtained properties close to the target properties. Board number 3 has MOE > 2600 MPa, MOR > 16 MPa and IB > 400 kPa. According to AS/NZS 4266, this particular board satisfies the minimum strength requirements for standard grade particleboard. Therefore, it is clear that particleboard with S – H configuration can achieve optimum properties for particleboard. However, the density for the optimal board (696 kg/m^3) remains slightly higher than the target density at 680 kg/m^3.

This optimal board also requires extra resin and moisture compared to industrial softwood board. Therefore, it was necessary to optimise the recipe to find the optimum processing parameters and obtain a board with optimum properties. Composite models for MOR, MOE, IB and density were developed incorporating the effects of process variables.

9.7 Optimisation of particleboard

As discussed earlier in Section 9.2, partial regression coefficients, which are required to develop a polynomial function (Equation 9.1) relating the tested property with significant parameters, were estimated using the least square regression method. MINITAB was used to calculate these coefficients incorporating design of experimental theories. The model presented here was developed using coded variables and should use only coded variables as factors. If the model is developed using un-coded variables, it can be used with variables with the same units. For instance, if the work uses the Celsius temperature scale, un-coded models cannot be used for data with Fahrenheit temperature scale. If the model is developed for coded date, then it is not dependent on the unit of the parameter but only on the parameter range.

Effects which generally have significant effects on most properties were considered for model development. Sometimes parameters with a less significant effect on a particular property may have a higher effect on another parameter. Therefore, during process optimisation, these effects become a variable. Hence, the effects with minor influence were also considered for the model. However, adding minor effects or non-significant variables may decrease the validity of the model by increasing the error. Therefore, further validation is essential to investigate the significance of the model.

The regression coefficients calculated for significant factors with respect to MOE, MOR, IB and density models are given in Table 9.4. These coefficients are calculated considering the coded variables.

Terms	Coefficient			
	MOE	**MOR**	**IB**	**Density**
Constant	678.642	-0.3656	-276.124	651.981
Moisture Surface	5.826	-0.0373	9.0487	-4.2454
Moisture Core	27.989	0.3848	22.5376	1.6358
Resin Surface	53.019	0.4602	28.1485	-1.2995
Resin Core	71.121	0.4182	23.4845	10.317

Table 9.4 Regression coefficients for significant factors with respect to responses

Incorporating these regression coefficients, the process model for each MOR, MOE, IB and density can be compiled as following equations:

MOE = 678.642 + 5.826*MS + 27.989*MC + 53.019*RS + 71.121*RC Equation 9.2

MOR = -0.3656 – 0.0373*MS + 0.3848*MC + 0.4602*RS + 0.4182*RC Equation 9.3

IB = -276.124 + 9.0487*MS + 22.5376*MC + 28.1485*RS + 23.4845*RC Equation 9.4

Density = 651.981 – 4.2454*MS + 1.6358*MC -1.2995*RS + 10.317*RC Equation 9.5

where,

MS = Moisture surface

MC = Moisture core

RS = Resin surface

RC = Resin core

9.7.1 Significance of Model

After generating the model equations to relate the dependent and independent variables, the validity of the model was checked experimentally by comparing the predicted values with the actual tested values.

Modulus of Elasticity (MOE)

Figure 9.1 compares the actual MOE values for the manufactured boards with the model predicted values. Equation 9.2 was used to generate the model predicted values by substituting the variables of each board into the equation. It shows that the process model represented by Equation 9.2 closely predicts the actual MOE of a particleboard.

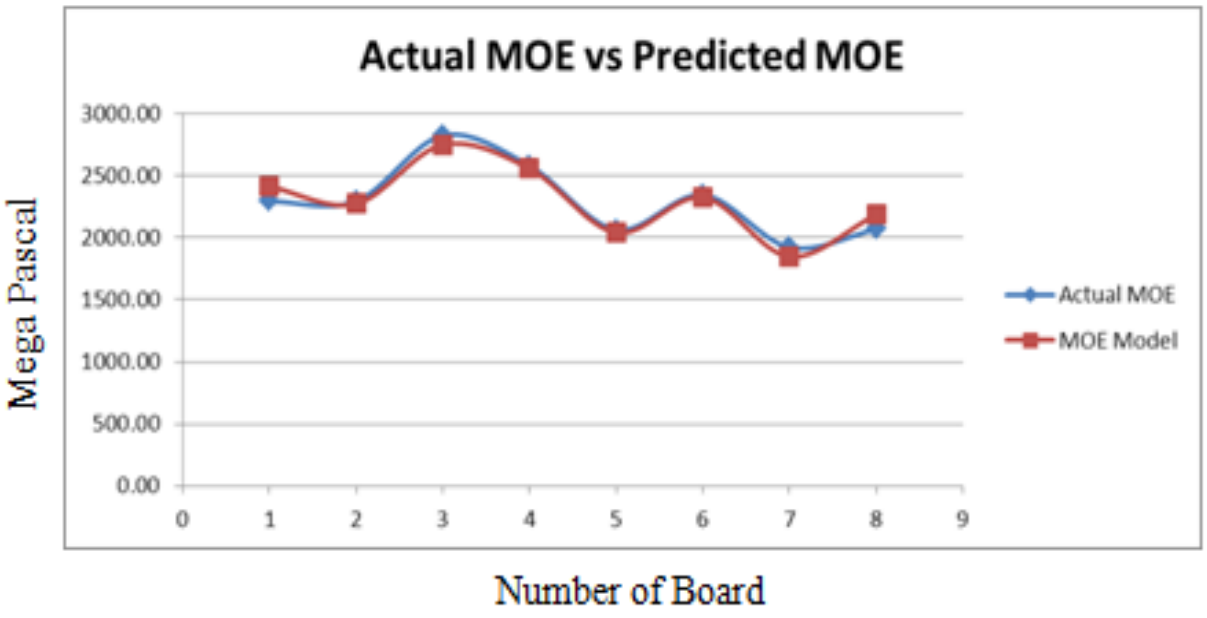


Figure 9.1 Actual MOE vs Predicted MOE by equation 9.2

Table 9.5 verifies the validity of the MOE model. As the MOE error column shows, Equation 9.2 predicts the actual MOE with less than 6 % variation. Almost 80 % of the boards produced an MOE error less than 5 %, with the exception of boards 1 and 8. This variation may be due to an error in the model or during the manufacture of the board. This level of precision confirms the validity and significance of the model.

Board no.	Actual MOE	Predicted MOE	MOE Error %
1	2294.39	2413.12	-5.17
2	2294.39	2273.44	0.91
3	2824.26	2750.03	2.63
4	2581.20	2557.65	0.91
5	2064.95	2041.40	1.14
6	2346.55	2325.60	0.89
7	1923.24	1849.01	3.86
8	2067.20	2185.93	-5.74

Table 9.5 Actual and model-predicted values for MOE

Modulus of Rupture (MOR)

As discussed earlier, a process model for Modulus of Rupture (MOR) was generated as Equation 9.3. In order to validate the significance of the model, as similar method to that for MOE was used. Figure 9.2 shows the comparison of experimental MOR and the MOR predicted by Equation 9.3.

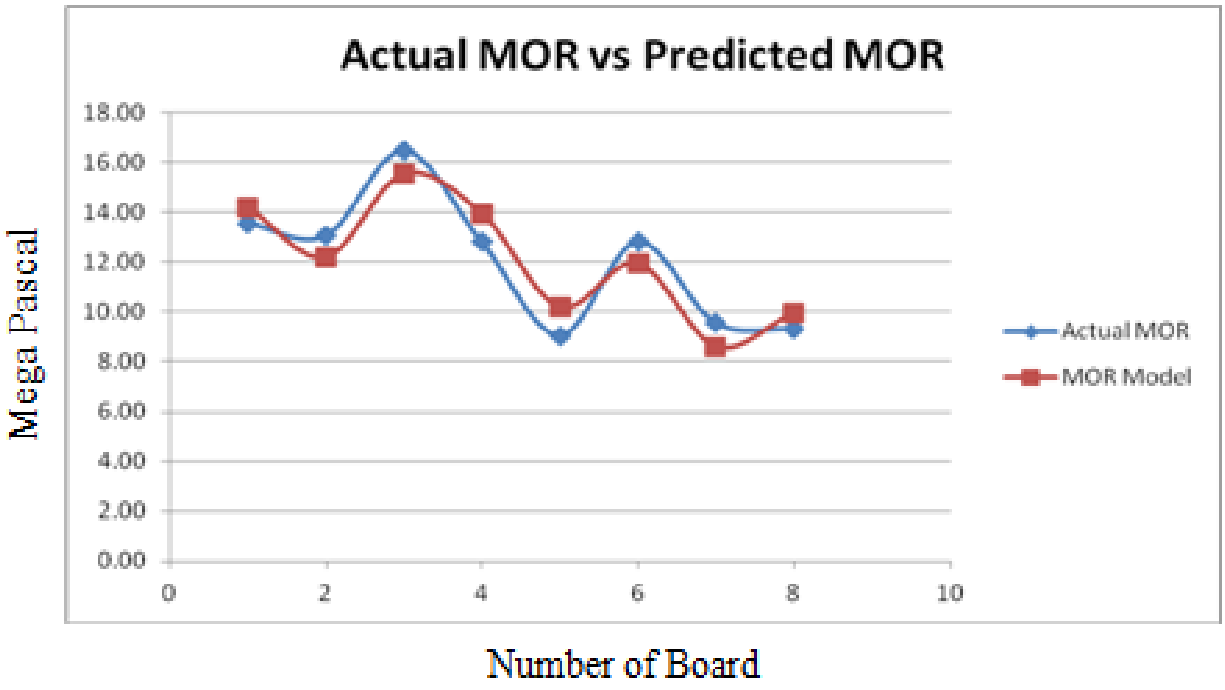


Figure 9.2 Actual MOR vs MOR predicted by equation 9.3

Figure 9.2 clearly demonstrates that the process model for MOR can precisely predict the actual MOR. Both actual and predicted MOR show similar trend lines and the predicted MOR values are almost identical to the actual MOR.

With the assistance of Table 9.6, the process model of MOR can be verified. From Table 9.6, it can be seen that Equation 9.3 can precisely predict the MOR with an error less than 13 %. Most of the boards have variations less than 10 %, with the exception of board no. 5. All these minor variations may be due to experimental error during particleboard production.

Board no.	Actual MOR	Predicted MOR	MOR Error %
1	13.56	14.21	-4.78
2	13.06	12.20	6.57
3	16.48	15.54	5.70
4	12.81	13.96	-8.98
5	9.04	10.19	-12.73
6	12.81	11.95	6.70
7	9.54	8.60	9.85
8	9.29	9.94	-6.98

Table 9.6 Actual and model-predicted values for MOR

Internal Bond (IB)

After generating the IB model equation to predict the IB of a particleboard, the validity of the model was checked by using a similar method as that for MOE and MOR. Table 9.7 shows the resulting IB values of the particleboards as well as the predicted IB values from Equation 9.4 and the error. It also shows that Equation 9.4 adequately predicts the IB of particleboard with less than 19 % variation. Over 80 % of boards attained a difference less than 14 % (board numbers 1 to 7 as seen in Table 9.7). Four of the boards (numbers 3 to 6) have variation less than 10 %. As the variation is very minor, the model is confirmed as able to predict the IB.

Figure 9.3 shows the comparison plot of actual IB and predicted IB, and it can be seen that the patterns for both plots are similar. Four points (1, 2, 6 and 8) have a greater variation between the actual and predicted IB. However, these variations had been justified in Table 9.7 that the model is tolerable to predict the IB of particleboard.

Board no.	Actual IB	Predicted IB	IB Error %
1	606.25	690.53	-13.90
2	631.25	559.28	11.40
3	906.25	865.91	4.45
4	643.75	671.78	-4.35
5	518.75	546.78	-5.40
6	731.25	659.28	9.84
7	393.00	352.66	10.27
8	443.75	528.03	-18.99

Table 9.7 Actual and model-predicted value for IB

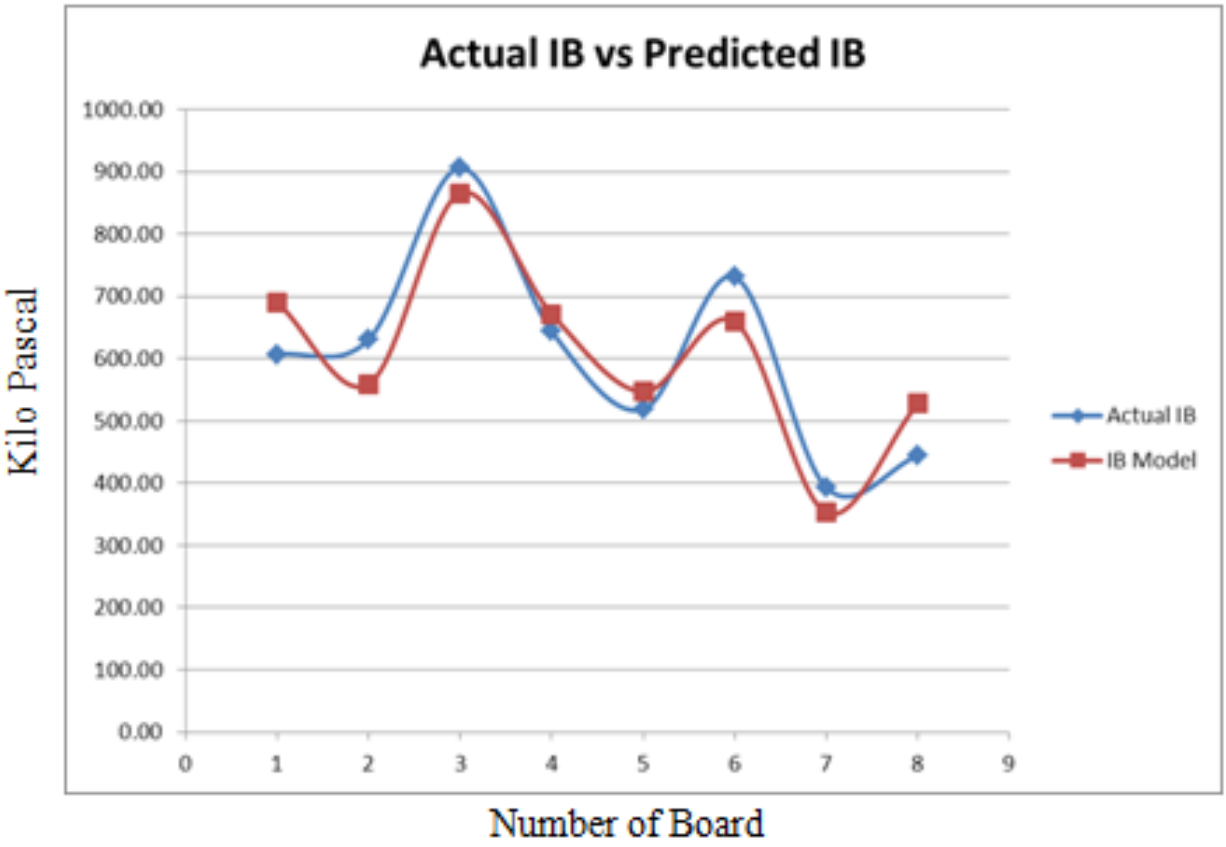


Figure 9.3 Actual IB vs IB predicted by Equation 9.4

Density

As process model of density was generated as Equation 9.5. Similar to the board properties discussed earlier, the significance of the process model for density was validated by comparing the experimental results with the model-predicted results. Figure 9.4 shows the comparison of actual density and predicted density. It can be seen that four of the actual densities (boards 1, 4, 5, 8) have significant differences from the predicted densities. Four of the actual densities (boards 1, 3, 7, 8) are higher than the predicted values. These variations are further compared in Table 9.8.

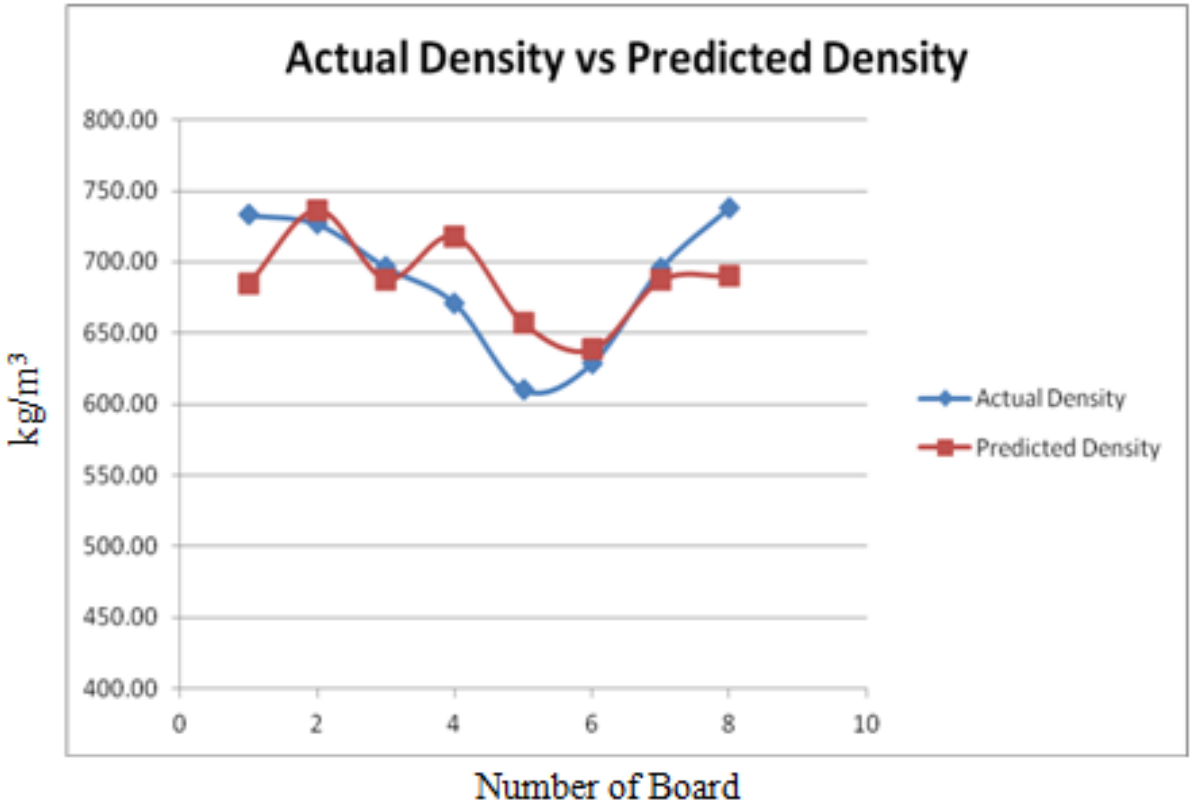


Figure 9.4 Actual Density vs Density predicted by Equation 9.5

The variations between the actual and predicted densities were compared and are tabulated in Table 9.8 as density errors in percentages. Table 9.8 shows that most of the boards showed minor variation of less than 8 %. These variations clearly justify the validity and significance of the process model.

All these process models were then further used for board optimisation. An optimal board was produced in the laboratory and analysed.

Board no.	Actual Density	Predicted Density	Density Error %
1	733.12	684.83	6.59
2	726.68	736.49	-1.35
3	696.39	687.89	1.22
4	670.95	717.92	-7.00
5	610.05	657.02	-7.70
6	628.63	638.44	-1.56
7	695.54	687.05	1.22
8	738.39	690.10	6.54

Table 9.8 Actual and model-predicted values for Density

9.7.2 Optimisation Plot

As discussed in Section 9.7.1, the process model for MOE, MOR, IB and Density were first validated. These process models were then used to find an optimised recipe or combination of variables for board optimisation. By using MINITAB, an optimisation plot was generated relative to the process models. The plot shows how the factors affect the predicted responses and allows the modification of factor settings interactively. The optimal board recipe can be produced by adjusting the factor settings to match the desired properties. Figure 9.5 below shows the optimisation plot generated from MINITAB.

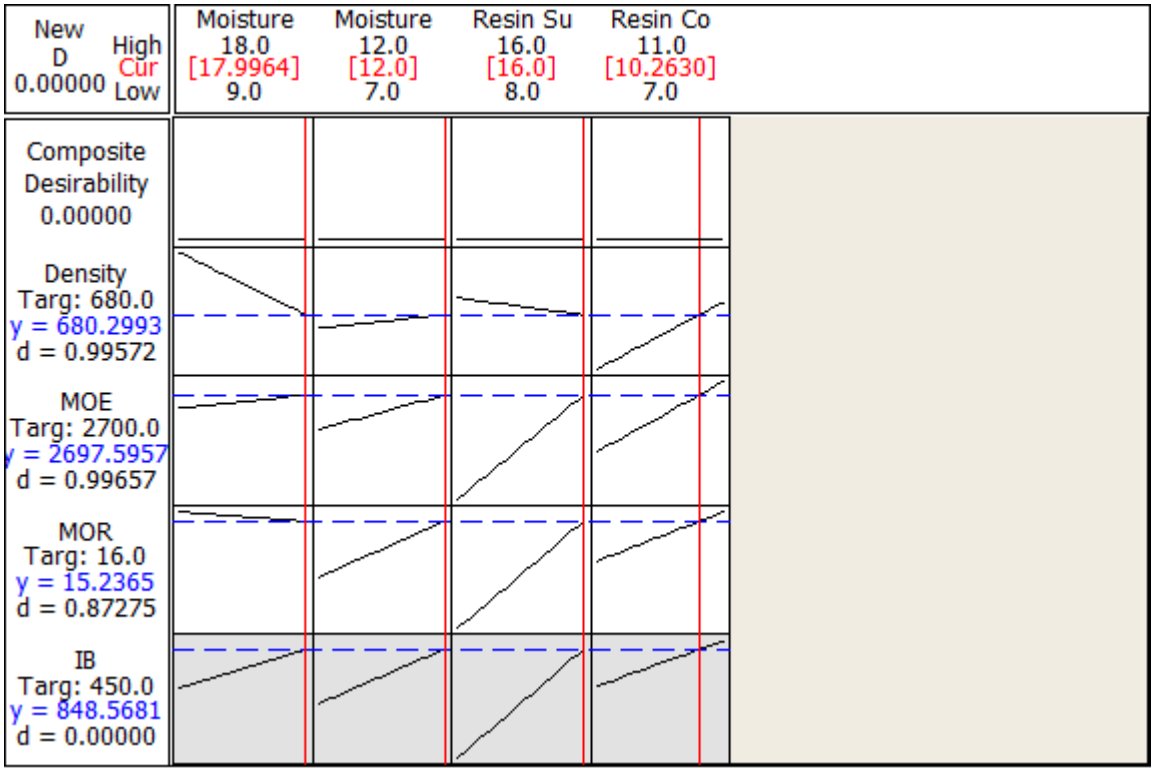


Figure 9.5 Optimisation plot for board optimisation

As seen at the left column in Figure 9.5, MINITAB shows the target values for each board property and also the predicted properties value, y, at the current factor settings. The numbers displayed at the top row show the current factor level settings (in red) and the high and low factor settings in the experimental design. The desirability, d, is also displayed in the left column with the response variables. The desirability values for each predicted response help to understand how close the predicted responses are to the target requirements. Desirability is normally measured on a 0 to 1 scale.

The relationship of variables with each response is clearly shown in each cell of the graph. For instance, the relationship of moisture surface with MOE, MOR, IB and Density can be seen in the second column in the figure. Increasing the surface moisture content increases the MOE and IB but MOR and density decrease. Therefore, in order to achieve an optimal board, it is important to recognize the relationship between the variables and board properties.

From Figure 9.5, it can be observed that optimal predicted properties can be achieved with the variables at moisture surface = 17.9964, moisture core = 12, resin surface = 16 and resin core = 10.2630. With these variable settings, the predicted density has desirability score of

0.99572 because the predicted response for density of 680.2993 is about 99.6 % of the way from the upper bound of 750 to the target of 680. MOE has a desirability score of 0.99657 because the predicted response for MOE of 2697.5957 is about 99.7 % of the way from the lower bound of 2000 and the target of 2700. For MOR, the desirability score is 0.87275. IB has the desirability score of 0 as the predicted IB value is not within the range of the upper and lower bounds. However, the predicted IB value at 848.5681 is sufficient for the tensile strength of standard board.

9.7.3 Validation of optimal board

After deciding the optimal variables using the process models, they were validated by comparing the properties of actual boards made in the laboratory with the predicted optimal board properties. Table 9.9 below shows the results of the comparison.

Properties	Actual results	Predicted results	Error, %
MOE	3142.16	2697.60	14.15
MOR	14.81	15.24	-2.9
IB	979.62	848.57	13.38
Density	689.33	680.30	1.31

Table 9.9 Validation of optimal board via comparison of actual and predicted board properties

The table shows that process models for each board property are sufficient to predict the actual board properties and also optimise boards. The resulting MOE is 3142.16 MPa, which satisfies Australian Standard requirement AS/NZS for particleboard. In comparison to the model predicted value of 2697.60 MPa, the MOE has a variation of 14.15 %. Although the error is slightly higher than expected, it is within the acceptable range and the resulting MOE value is even better than the predicted value. IB has an error of 13.38 %. However, the

resulting IB value at 979.62 kPa is still much higher than the predicted value. This IB value will have no effect on satisfying the AS/NZS requirement.

The process model of MOR and density predicts the actual board properties, and both MOR and density have minor errors of -2.9 % and 1.31 % respectively. Although the resulting MOR is slightly lower than the predicted value, it is adequate to satisfy the AS/NZS requirement. The actual MOR was found to be 14.81 MPa while the density of board has been optimised to 689.33 kg/$m^{3,}$ at which it is sufficiently close to the requirement of AS/NZS.

9.8 Summary

Chapter 9 has presented the final optimization of board properties to achieve a satisfactory particleboard which is economical and uses a large quantity of hardwood saw- mill residue.

A factorial experimental design was used to develop a process model to predict the required board properties as a function of process and material variables. The developed model was then used to identify the optimum process and material variables. This optimized board was made in the laboratory and was also used to test the validation of the process model.

Table 9.10 shows the optimized board with the respective properties. The process adopted so far has utilized a statistical approach in solving the basic research problem. It was also considered important to understand the chemical process of resin curing which produces the observed behavior. Chapter 10 attempts to understand the underlying reasons for the observed behaviour using surface studies of particleboard.

Chapter 10

SURFACE STUDIES AND THERMAL ANALYSES OF HARDWOOD AND SOFTWOOD COMPOSITES

10.1 Overview

On the basis of the research findings presented in previous chapters, it was suspected that particleboard produced from hardwood and softwood residues has a different board structure and curing reaction of UF resin. Therefore, further studies were carried out to explore this phenomenon. GADDS (General Area Diffraction Detector System) and Thermogravimetry Analysis (TGA) techniques were used for this investigation.

10.2 Introduction

With the application of GADDS, it is possible to obtain the different crystalline structures of hardwood and softwood particleboards. GADDS is the most advanced X-ray diffraction system in the world. The core of GADDS is the high performance two dimensional (2D) detectors (HI-STAR) which can simply collect data 10^4 times faster than a point detector and also about 100 times faster than a linear position-sensitive detector. From polymers to metals, this system can collect data at a higher resolution than a conventional system. This makes collecting data on large grain materials especially easy. A better picture of the properties of new materials being developed can be investigated easily in greater detail.

Thermogravimetric analysis (TGA) is a type of analysis that determines the changes in weight of a sample in relation to changes in temperature. It is commonly used in research to determine the characteristics of materials. The degradation temperature of materials and the levels of inorganic and organic components in materials can also be determined via TGA. It characterizes the composition and thermal stability of materials under a variety of conditions and examines the kinetics of the physico-chemical processes occurring in the sample. The composition of hardwood and softwood can be simply found through TGA.

10.2 Objectives

As discussed earlier, General Area Diffraction Detector System (GADDS) and Thermogravimetric analysis (TGA) were selected to determine:

1. surface analysis using GADDS mapping
2. composition analysis of hardwood and softwood using TGA

10.3 Experimental Program

10.3.1 General Area Diffraction Detector System (GADDS)

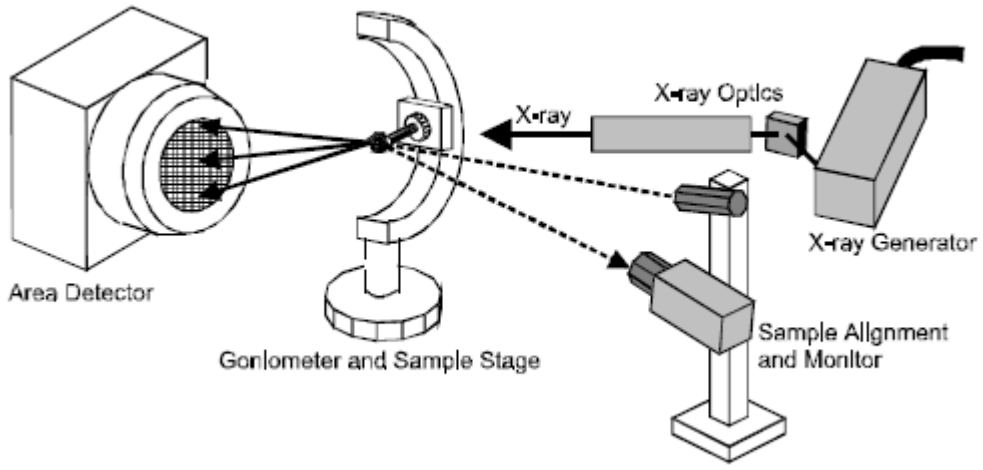


Figure 10.1 Diagram of GADDS instrument

Figure 10.1 shows a typical GADDS system used in the laboratory. As seen in Figure 10.1, a GADDS system normally consists of five major units:

- an X-ray generator to produce X-rays
- X-ray optics to condition the primary X-ray beam
- a goniometer and sample stage to establish and maneuver the geometric relationship between primary beam, sample, and detector
- a sample alignment and monitor to assist users in positioning the sample into the instrument's centre and in monitoring the sample state and position
- a detector (HI-STAR area detector system) to intercept and record the scattering X-rays from a sample and to save and display the diffraction pattern into a two-dimensional image frame

In this research, GADDS was used to map the composites and to check uniformity of the samples on the surface. GADDS was selected as it contains far more information than conventional powder X-ray diffraction (XRD) methods. It can collect diffraction data in a large 2θ range simultaneously without sample or detector movement. In addition, a conventional X-ray diffraction cannot be used for mapping large samples and hence cannot depict the distribution of a particular species in the sample.

GADDS facilitates the examination of sample areas on micro-spatial length scale by using the X-ray optics to focus the excitation beam to a small spot on the sample surface (Bergese et al. 2001, Flemming 2007). GADDS is a micro X-ray diffraction system employing a high sensitivity 2D area detector. It is extremely useful in recognising the phase changes in scales and in assemblages within secondary formations. This is one of the few techniques that can identify the phases directly in an in-situ sample, without separating or crushing the sample. One of the main features of the GADDS system is a high performance 2D detector (Flemming 2007; Manual 2004; 2007, He 2004). The 2D detector captures the whole or a large portion of the diffraction rings that can be integrated over a selected area and the resulting image and integrated diffraction pattern contains more information than traditional powder patterns. GADDS has the capability to identify phases accurately and to analyse texture quantitatively at micron level scales, which is not possible using typical 1D detectors (Manual 2004, 2007). The laser video sample alignment system allows the user to align the intended measurement spot on the sample precisely with respect to the X-ray beam. The

motorized XYZ stage can move the measurement spot to the instrument centre and map many sample spots automatically. The video microscope can record the image and position of each cell of the material during the data collection. Phase identification can be done by integration over a selected range of angles 2θ and ψ. Most importantly, θ–θ geometry in combination with XYZ sample stage in GADDS permits a wide range of samples such as whole rocks, cut sections, and polished sections (Flemming 2007). The GADDS instrument is relatively new and to date has not been used extensively for wood samples.

10.3.2 Thermogravimetry (TGA)

The Pyris 1 TGA is used in the laboratory for the investigation of hardwood and softwood composites. The Pyris 1 TGA is made up of two major components:

- a microbalance and
- a furnace element

The microbalance operates as a high gain electromechanical servo system that allows the detection of weight changes as small as 0.1 g, with a maximum capacity of 1300 mg. The null balance design uses a servo-controlled torque motor to automatically compensate for weight changes in the sample. When a sample is placed in a sample pan and the pan is on the hang down wire, the beam that supports the sample pan deflects. A beam position detector measures the deflection with an optical sensor and uses a current to return the beam to its original position. The amount of current necessary to maintain the system in the "null" state is directly proportional to the weight change in the sample.

Two furnace options are available: standard and high temperature. The standard furnace allows operation of the Pyris 1 TGA from sub-ambient to 1000°C. The furnace uses a micro-furnace that has a low thermal mass for quick cooling and equilibration. The platinum heater element acts as a resistance temperature detector under tight feedback control. The furnace detects its own temperature and supplies power to heat the sample. Accurate program temperature control reduces overshoot to 0.2°C at 100°C/minute which results in stepwise analysis precision. The sample temperature is sensed by a chromel–alumel thermocouple. Most standard polymer applications are routinely performed with the standard furnace (Pyris User Manual).

The high temperature furnace allows operation of the Pyris 1 TGA from 50°C to 1500°C. This furnace uses platinum / 30 % rhodium heating elements and can be operated at scanning rates of 0.1°C/min to 50°C/min. The high temperature furnace provides excellent temperature control and precision (±5°C) for even the most demanding applications. A sensitive platinum/10 % rhodium – platinum thermocouple in close proximity to the sample measures the sample temperature during analysis. The high temperature furnace is used for applications such as ceramics, metals, and geological studies.

Autosampler is one of its most advance features designed for the laboratory and the quality control or quality assurance environment. The autosampler enables samples to be prepared and run during the same shift. Using the Pyris Player feature of Pyris Software for Windows, play lists can be created that will run the samples in the autosampler tray and analyze the data automatically.

10.4 Materials and methodology

10.4.1 Settings of GADDS

Samples were analysed using a Bruker AXS D8 - Discover system with GADDS using Cu-Kα radiation and operated at 40 kV and 40 mA. The sample distance was maintained at 15 cm giving a 2θ range on the 2D detector of 34 °. In this experiment, we set Ω = 20 ° and θ = 40 °. The collection time for each spot was 600 seconds. After careful spot analysis, areas of specific interest were selected for mapping with a collection time of 1200 seconds. In both spot analysis and mapping, a collimator of size 0.2 mm was used. A 0.2 mm collimator scans an elliptical pattern of vertical diameter 0.2 mm and horizontal diameter 0.65 mm across the sample at each selected point. The instrument was calibrated using the NIST standard of Al_2O_3.

10.4.2 Settings for TGA

Samples at approximately 58 mg were prepared from hardwood and softwood respectively. Experiments were performed in a Simulated Thermal Analyser 6000 (STA 6000) at 10 °C/ min in a temperature range of 30 – 1000 °C. These heating rates, namely 10, 20 and 30 °C/min were used to understand the effect of heating rate on the thermal kinetics and emissions of the samples. For each experiment, weight loss of the samples was determined as a function of temperature. The output of the instrument gives the TG (Thermo gravimetric) curves as well as the derivative curves (DTG) of the sample.

10.5 Results and discussions

10.5.1 GADDS

Li et al. (2009) found that XRD is widely used in material characterization, particularly to determine crystallographic structure and orientation, crystalline quality and residual strain of the crystalline structure as well as the chemical composition of natural and manufactured materials. Several studies have been done to study the characterization in wood by using XRD (Lotfy et al. 1974; Tanaka et al. 1981; Dwianto et al. 1996; Kubojima et al. 1997).

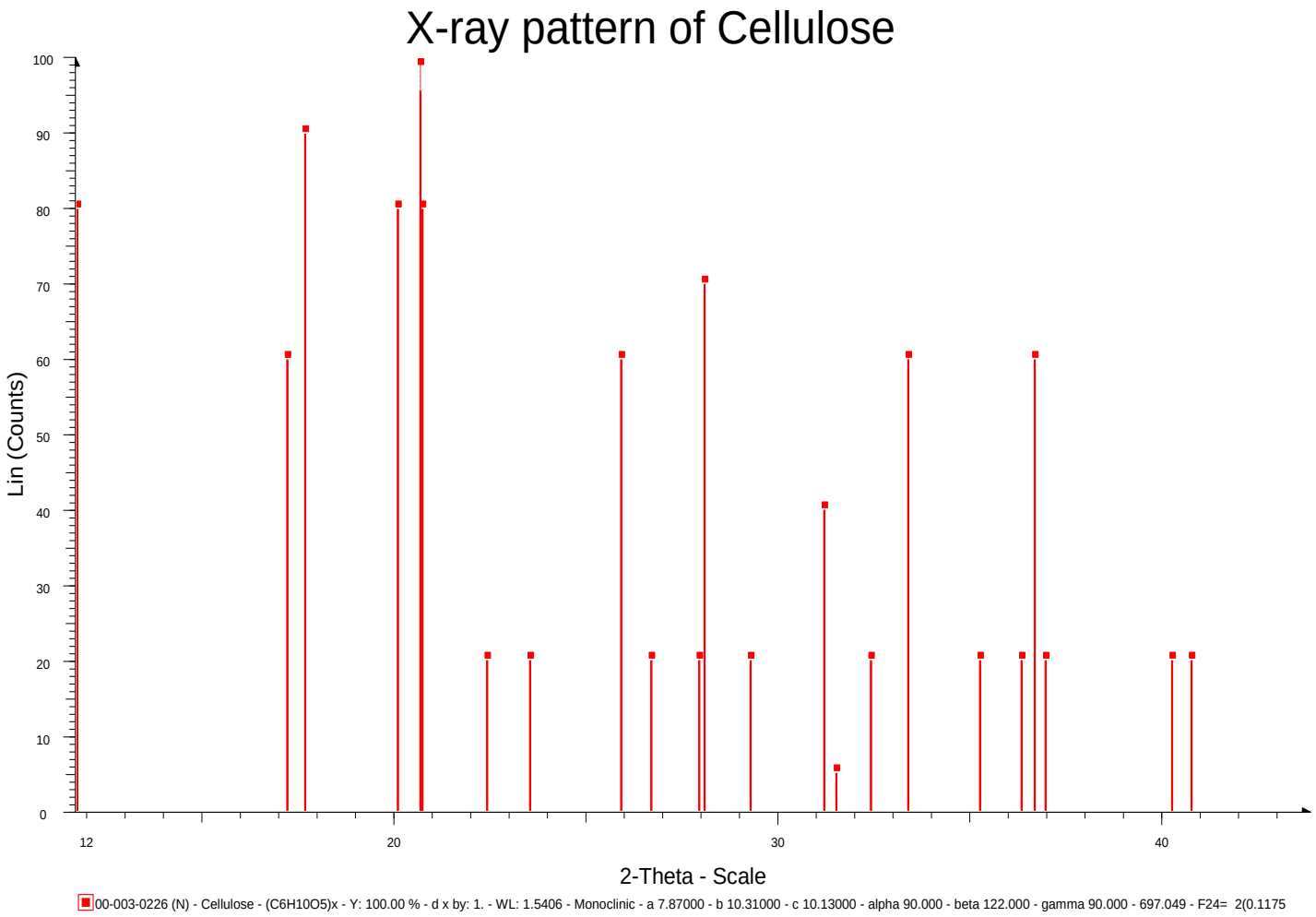


Figure 10.2 X-ray diffraction pattern of cellulose obtained from ICDD database

Figure 10.2 shows the typical diffraction pattern of cellulose (ICDD database). Wood consists of cellulose, hemi-cellulose, lignin and other components. In this study, the diffraction patterns of cellulose were taken as the hypothesis and a novel technique was applied to study differences on the surface of the particleboard (GADDS mapping – refer Experimental section page for more details).

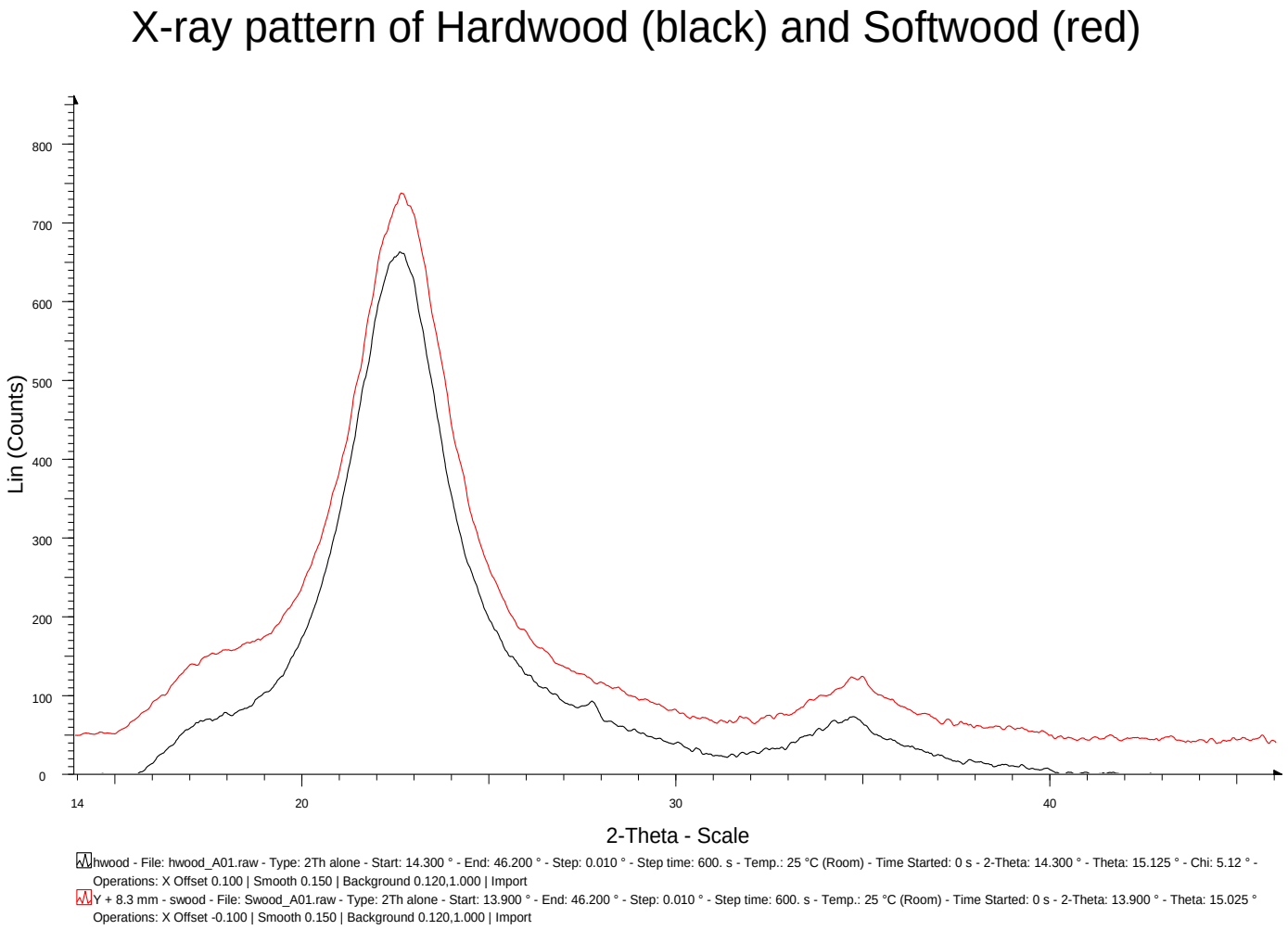


Figure 10.3 X-ray patterns of Hardwood and Softwood

Figure 10.3 shows the X-ray patterns of hardwood and softwood composites respectively. It is important to note that the X-ray mapping was only done for the surfaces of hardwood and softwood composites. In comparing hardwood and softwood in Figure 10.3, it can be noted that hardwood and softwood produce a similar X-ray pattern to cellulose. This shows that both composites reach a major peak intensity at the diffraction angle at $2\theta = 23^{o}$ and two secondary peaks at $2\theta = 17.5^{o}$ and $2\theta = 35^{o}$. An obvious decrease in the intensity of major peak and secondary peak at $2\theta = 35^{o}$ can be observed for both hardwood and softwood in Figure 10.3. After the secondary peaks at $2\theta = 17.5^{o}$, there is a steep increase to the major peak intensity for both hardwood and softwood. However, there is a minor distinction at the diffraction angle $2\theta = 28^{o}$. It can be clearly seen that there is minor peak intensity for hardwood at $2\theta = 28^{o}$.

Lazarescu et al. (2010) established that X-ray diffraction is capable of measuring the interior properties of wood. X-ray diffraction (XRD) with a wide angle was utilised by Marashdeh et al. (2011) to investigate the crystallographic structure and orientation of different types of Rhizophora spp. particles. XRD diffraction analysis indicated that the crystallinity of binderless particleboard decreases as the particle size decreases. This clearly shows that hardwood is more crystalline (χ_d = 97 %, CrI = 89 %) than softwood (χ_d = 89 %, CrI = 77 %). Crystallinity can be simply calculated using two methods using the equations below:

$$\chi_d = \left[1 - \frac{S_a}{S_a + S_{cr}}\right] x100$$ Equation 10.1 (Focher et al. 2001)

where,

χ_d = crystallinity

S_a = armophous integrated area

S_{cr} = sum of the area of crystalline peak

$$CrI = \left[\frac{I_{002} - I_{am}}{I_{002}}\right] x100$$ Equation 10.2 (Segal et al. 1959)

where,

I_{002} = intensity for the crystalline portion of biomass (i.e., cellulose)

CrI = degree of crystallinity

I_{am} = maximum intensity of the amorphous peak

Much research has been carried out to investigate the applicability of XRD to wood species. For example, a study of the structure of wood cells was completed by Paakkari et al. (1984). X-ray diffraction method was used to determine the values of the mean microfibrillar helical angles and to estimate quantitatively the amount of crystalline cellulose in the various cell wall layers of wood fibres.

Borysiak et al. (2005) carried out an investigation of the supermolecular structure of pine wood after mercerisation by sodium hydroxide. X-ray diffraction was used for this study. X-

ray diffraction patterns of unmodified and mercerised pine wood were compared. It was found that polymorphic transformation of cellulose in pine wood depends on the conditions of chemical treatment (concentration of NaOH and time of treatment). The mercerisation process caused an overall decrease in the degree of crystallinity of pine wood.

These previous studies show that X-ray diffraction analysis is suitable to obtain the structure and interior properties of wood. Whilst a detailed chemical analysis was beyond the scope of this work, it was used to understand the general quality of the boards and also identify areas which require further research. A further study using TGA was considered essential to investigate the differences between hardwood and softwood composites at diffraction angle $2\theta = 28^{o}$.

10.5.2 TGA

Figure 10.4 shows the Thermogravimetric (TG) and Derivative of Thermogravimetric (DTG) results for the hardwood particles. As Figure 10.4 shows, the DTG curve indicates four regions of weight loss. The first loss of weight of the hardwood takes place in the region between 47 – 200^{o}C with the weight loss at about 3.3 %. The second region is between 200 – 330^{o}C with the weight loss of approximately 26.7 %; the third region is between 330 – 390^{o}C with the weight loss of 64.5%. The fourth region can be found between 390 – 800^{o}C with the weight loss of 63.2 %.

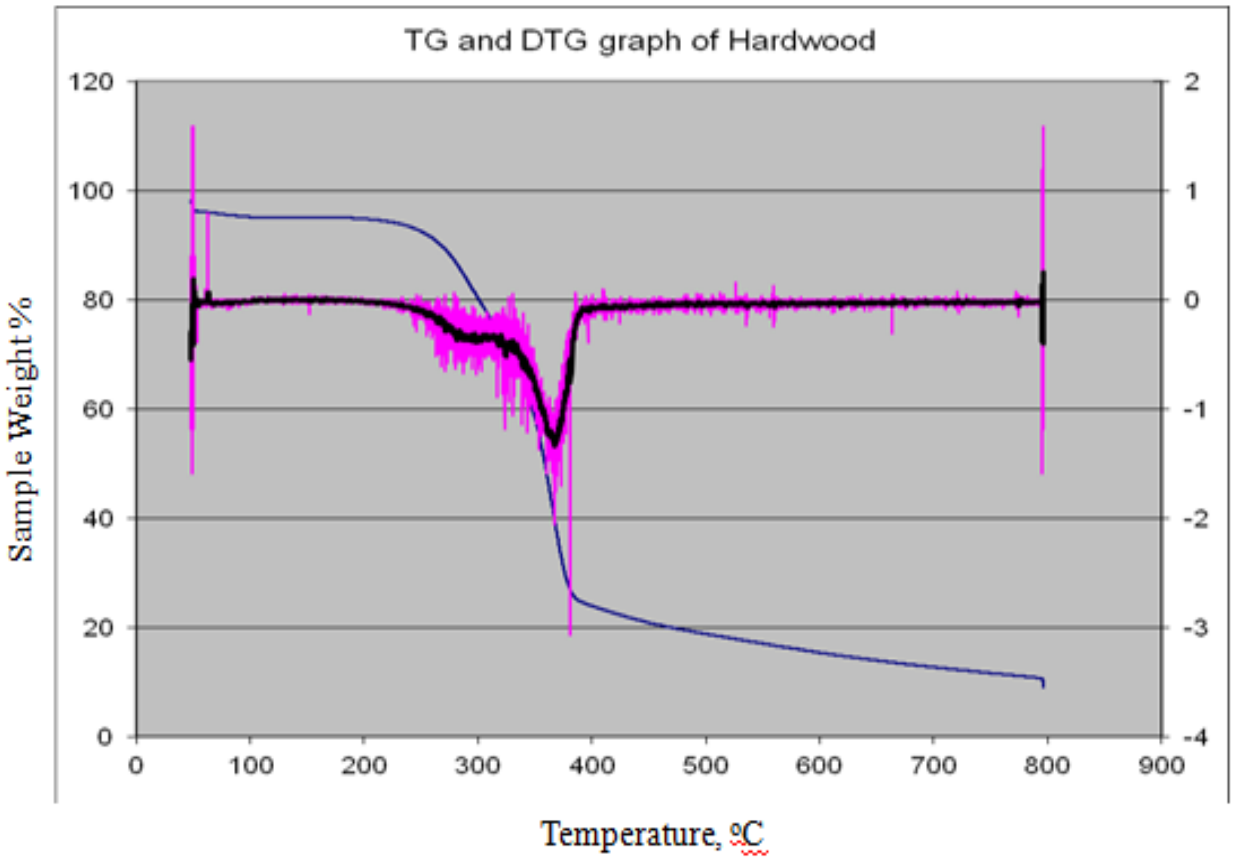


Figure 10.4 TG and DTG graph of Hardwood

TGA has also been used for softwood particles. Figure 10.5 shows the TG and GDT graph of softwood particles. Similar to hardwood particles, there are four regions of weight loss for softwood particles. The first loss of weight of softwood takes place in the region between 47 – 200^{o}C with the weight loss at about 3.8 %. The second region is between 200 – 330^{o}C with the weight loss of approximately 24.4 %; and the third region is between 330 – 390^{o}C with the weight loss of 57 %. The fourth region can be found between 390 – 800^{o}C with the weight loss of 50.7 %.

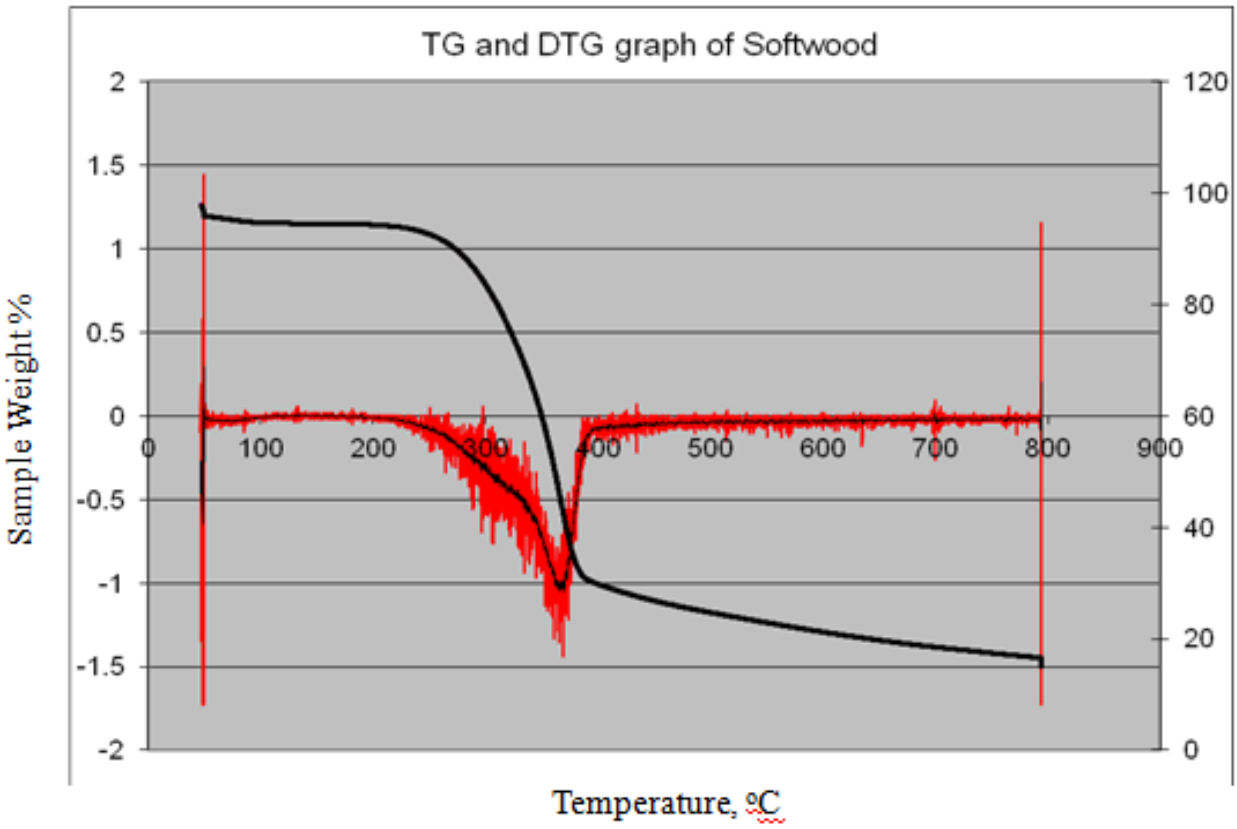


Figure 10.5 TG and DTG graph of Softwood particles

In comparing hardwood and softwood particles, it is noted that both hardwood and softwood show the largest weight loss at the third region. However, hardwood particles result in a higher weight loss than softwood in this region, being 64.5 % and 57 % respectively.

The temperature of the peaks in the DTG curves depends on the heating regime (Vlaev et al., 2003). From the DTG curves in Figure 10.4 and Figure 10.5, the peak temperature and burnout temperature can be found. The peak temperature represents the place where the rate of weight loss is at a maximum and it is mainly used in the assessment of combustibility or ignition. The burnout temperature shows the temperature where sample oxidation is completed (Sonibare et al., 2005). Comparing both hardwood and softwood, it is found that both particles have similar peak and burnout temperatures at approximately 365^{o}C and 400^{o}C respectively.

According to Gao et al. (2006), thermal degradation of hemicelluloses occurs first, and then cellulose decomposes. Thermal degradation occurs at lower temperatures, because of dehydration, oxidation, the evolution of carbon dioxide and the formation of carbonyl and

carboxy groups. At higher temperatures, rapid volatilisation takes place. Volatile and flammable products are released at this temperature. In contrast to cellulose and hemicelluloses, decomposition of lignin takes longer by char oxidation (Gao et al., 2006; Nassar, 1999). These statements clearly explained the decomposition mechanisms of softwood and hardwood particles.

Although the study of TGA on hardwood and softwood particles did not illustrate any significant distinction between hardwood and softwood, it may good practice for a better understanding of the decomposition of hardwood and softwood. By knowing each decomposition temperature, it may be easier to control the pressing temperature during particleboard production in order to maintain board properties or prevent any decomposition of wood particles.

A further study was completed in collaboration with the School of Applied Sciences at RMIT University to examine the mass loss of composites to establish thermal stability. Particleboard made of hardwood sawmill residue and softwood was prepared and compared with three commercial particleboard composites (‘blue’, ‘white’ and ‘brown’).The results are displayed in Figure 10.6.

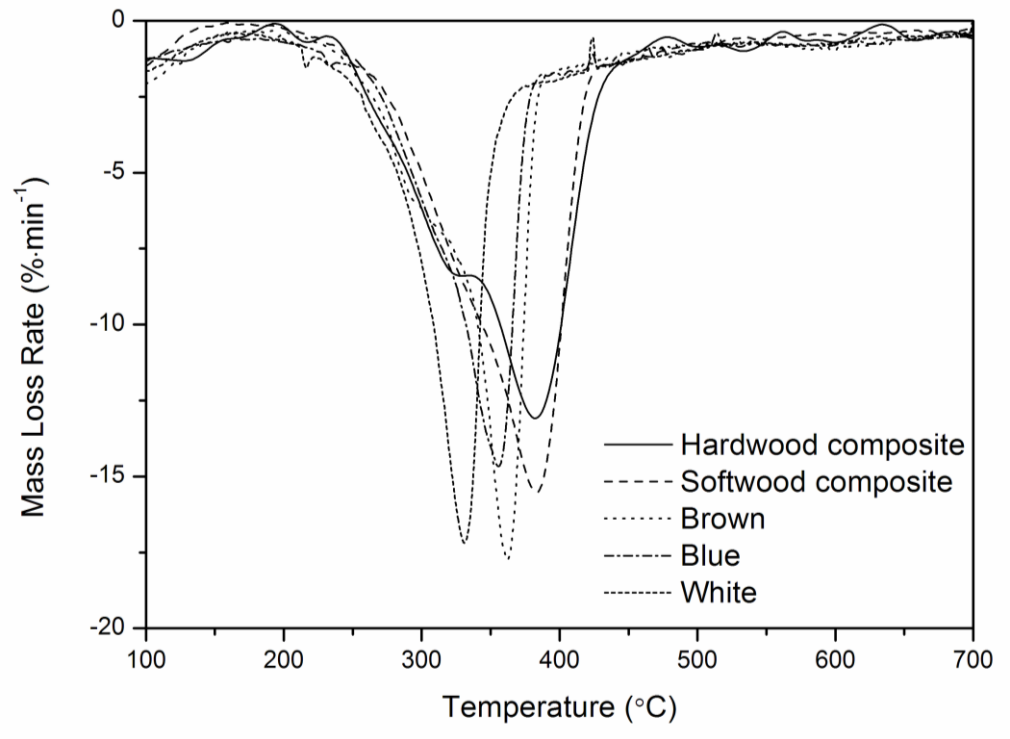


Figure 10.6 Derivatives mass loss curves of wood-resin composites

Both laboratory manufactured boards display a maximum degradation temperature of 385 ^{o}C, higher than brown (364 ^{o}C), blue (358 ^{o}C) and white (333 ^{o}C). This behaviour is most probably due to an increased amount of resin and the subsequent degree of cross-linking within the prepared composites (Athawale and Alhousami 2008). Back (1967) observed that effective cross-linking can stabilise the cellulose structure by providing additional bonds that are stronger than hydrogen bonds. These stronger bonds must be broken in order for the degradation mechanism of cellulose to occur. More effective cross-linking within the softwood- and hardwood-UF composites produces thermal stability superior to commercial particleboard. Therefore, it is confirmed that hardwood and softwood particleboard produced in the laboratory have better thermal stability than commercial particleboard.

A comparison with the findings from research indicates the following. Osvald and Reh (1992) and Reh et. al (1993) used TG analysis in particleboards treated with $NH4H_2P0_4$. Weight loss of the particleboards decreased with increasing level of the retardant application and this was evident in the whole temperature interval. The reason was probably the beginning of the endothermic degradation of the retardant used connected with the releasing H_20 and NH_3.

Vovelle et al (1983) compared the thermal stability of painted and unpainted particleboards by means of TGA. Thermal degradation of particleboards started around 250 °C and the mass loss rate reached maximum for a temperature close to 300 °C. This maximum corresponds to the degradation of cellulose. For all painted particleboards, the beginning of mass loss is observed at a higher temperature, demonstrating that paints are more stable than wood. In painted particleboards, only a fraction of the initial mass of paint is gasified and the proportion of solid residue remaining at the end of the reaction could be as high as 70 %.

Panayotov (1996) compared the TGA data of fir wood and wood treated with three commercial fire retardants containing nitrogen, phosphorus or chloride. He found that the modified wood of its mass higher than the natural fir wood, and consequently it was more resistant to thermal destruction. He also observed in the thermogram of the modified wood that the rate of formation of the volatile products reaches its maximum at a lower temperature compared with natural fir wood.

The findings of these previous researchers indicate that TGA analysis can be used to establish the thermal degradation of boards, which gives an indication of the integrity of the boards.

The proposed new product therefore has the expected board stability for general purpose particleboard.

10.6 Summary

Whilst it was worthwhile to conduct surface studies and thermal analysis of hardwood and softwood composites, these methods were unable to confirm the nature of the curing of resin. Major findings of the chemical analysis are given below:

- Through GADDS, a minor differentiation of hardwood and softwood board surfaces can be observed at the diffraction angle $2\theta = 28^{o}$ in Figure 10.3. Further studies can be continued to advance the research to cover resin curing in the boards.

- Decomposition rates, temperatures and patterns of hardwood and softwood have been found with TGA. This method establishes a better understanding of the composition of hardwood and softwood. By knowing the composition of each hardwood and softwood, the hot pressing temperature can be adjusted so that board properties are optimized.

- Analysis of the thermal stability of the laboratory-manufactured particleboard and a comparison with commercial products confirm that the cross-linking of the hardwood-softwood product is superior to the commercially-available products.

10.6.1 Future recommendations

The surface analysis and composition studies of particleboard are beyond the scope of the present research. Hence, these are recommended as topics for future work. The following are some of the techniques that could be used in future studies to conduct reverse engineering and optimize the contents of particleboard.

1. Find the curing point of composites using a suitable thermal analysis technique (Differential Scanning Calorimetry)

2. Map the surfaces of composites by FTIR and Hot stage - image processing (softwood and hardwood composites)

3. Conduct a reverse engineering study of the degree of Urea Formaldehyde resin curing and compare the properties of softwood and hardwood-based composites

Chapter 11

CONCLUSIONS

11.1 Conclusions

The possibility of using hardwood residues as particleboard furnish has been investigated in this study with specific attempts made to explore mixes of hardwood residue and softwood flakes in the manufacture of general purpose particleboard, which satisfies AS/NZS standards. Four main series of experiments were conducted using the theory of experimental design. Important parameters affecting properties of particleboard were identified. Further, process models were developed to predict modulus of rupture (MOR), modulus of elasticity (MOE), internal bond (IB) and mean density of particleboard made out of mixtures of softwood and hardwood. These models were used to optimize the processing and material variables of particleboard and an optimum mixture design for making particleboard with 45 % hardwood residue has been developed. This chapter presents a consolidation of knowledge arising from the work reported herein.

11.1.1 Conclusions from the investigation in relation to significance of the project

- Studies by Nirdosha (2006) indicated that hardwood residues may be a suitable raw material for particleboard production. Hardwood particleboard was found to require longer pressing time and also higher density than commercial board products. Further

investigation was identified as essential to improve and optimize the application of hardwood residues in the wood panel industry.

- Hardwood residues are currently treated as waste at sawmills with global waste persistently increasing. In Australia, there is a significant amount of scrap material generated by sawmills and other wood processing factories. 200,000 m^3 from saw logs and 300,000 m^3 from pulp logs are considered as hardwood residue or wastes. Therefore, it is worthwhile to explore hardwood residues as raw materials for particleboard production.

- The price of a tonne of hardwood residues may range from $1.50 to $30. Economic analysis of the particleboard production process clearly indicates that the higher resin consumption is offset by the low cost of raw materials, except in the situation where the cost of hardwood residue escalates to be similar to that of softwood flakes.

- The new products will also offer significant environmental benefits which can overcome the additional costs incurred in using 25 % more resin for hardwood particleboard production.

11.1.2 Conclusions of the literature review

- Production of particleboard had been reviewed to develop a better understanding of the particleboard production process. Process parameters that need to be considered were also identified.

- The mechanical properties of particleboard (MOE, MOR and IB) are the main strength properties that need to be achieved to satisfy the requirements for general purpose particleboard. MOE and MOR are most likely dependent on the surface layers while IB is dependent on the core layer.

- MOR and MOE of particleboard are strongly dependent on various parameters, such as board density, particle configuration and orientation, material types and processing conditions.

- MOR and MOE of particleboard can be improved by increasing the compaction ratio as well as by increasing the length/thickness ratio for the same wood species. Most studies have reported an increase in MOR and MOE with increasing board density, as the compaction ratio increases the bending strength of board.
- IB is one of the very important properties of particleboard that is dependent on the core layer. Moisture content in the core and particle size within the core layer are important factors that need to be controlled to produce better IB properties.

11.1.3 Design of experiments for product development

- Design of experiments (DOE) using partial factorial design was identified as a technique to organize experiments with multiple variables for this investigation.
- 2^k fractional factorial design and the method of data collecting and analysis for experiments with multiple variables have been discussed.
- ANOVA was identified as a suitable technique for identifying significant parameters.
- Various researchers in the past have used the above techniques to develop new materials or to develop experiments with new materials and to optimize processes.

11.1.4 Method of making particleboard in the laboratory

- Chapter 5 discussed the methods used to produce particleboard in the RMIT laboratory. Methods and equipment were developed and preliminary trials were conducted.
- A spreadsheet was developed for the mixture design of particleboard using different raw materials and resin loadings. With the multiple variables considered in particleboard production, each single board produced may obtain different combination of variables setting in accordance to the screening test generated from MINITAB. Mix design is essential to ensure the right proportion of wood particles, resin and water during production.

- The study showed that particleboard has to be pre-pressed using a cold press before hot pressing to achieve improved strength properties. Pre-compaction of particleboard can be achieved with cold pressing.
- Testing methods for MOR, MOE and IB have also been clearly explained in this chapter. The testing equipment was designed according to Australian Standard AS/NZS.

11.1.5 Series 1: Particleboard production using mixtures of hardwood residue and custom flaked softwood

The first experimental design comprised 33 boards manufactured in the RMIT laboratory. Following are the findings of these experiments:

- None of the 33 boards met the requirement of AS/NZS general purpose particleboard.
- Pressing temperature and pressing time were found to be significant factors affecting particleboard properties as both are essential for resin curing and enable stronger bonding within particles.
- Higher hardwood proportion in the surface results in a higher strength of particleboard. Therefore, it is suspected that particle size in the surface layers significantly affects the bending strength of particleboard.
- A uniform moisture distribution was used throughout the surface to core layer of particleboard. Uniform moisture distribution over the particleboard was observed to cause spring-back or thickness swelling due to the excess moisture trapped within the board.
- Compared to hardwood particleboard, particleboard produced from mixtures of hardwood residue and custom flaked softwood has lower optimal strength properties. The board density remains higher than the target density.

11.1.6 Series 2: Particleboard production using mixture of hard/softwood in core layer only

Due to the unsatisfactory results from the first series of experiments, another series of particleboards was made with some adjustment of variables after the review of previous results. Two hypotheses were established as below:

- Hypothesis 1: Hardwood particles in the surface layer contribute to bending strength
- Hypothesis 2: Non-uniform moisture distribution over surface and core layers will improve board properties

Following are the findings in relation to the hypotheses for this series:

- An obvious improvement was observed in this series of boards as compared to the results of series 1. However, none of the boards met the requirement of the AS/NZS standard.
- Moisture is a critical variable that needs to be controlled carefully for better mat compaction as moisture is the main medium for heat transfer from surfaces to the core. Non-uniform moisture distribution (high surface moisture content; lower core moisture content) results in better board properties as it contributes to a shorter press cycle by accelerating the heat transfer over the board for resin curing without any excessive trapping of moisture.
- The acidity of hardwood particles disproves the hypothesis that hardwood particles only in surface layers contribute to bending strength. UF resin in particleboard production cures faster in acidic conditions. Therefore, with only hardwood particles in surface layers, the fast curing of surfaces would result in over-curing of the layers, which would degrade the glue bonding between particles.
- Higher resin content in core layer would result in better IB strength as resin is the main ingredient that creates permanent inter-particle bonds in the particleboard.
- Higher proportions of hardwood in the core would result in a better tensile strength. This is due to the acidity of hardwood particles. A higher proportion of hardwood in the core will lead to a faster curing in the core layer. Since the core layer might take

longer to reach a sufficient resin curing temperature, the acidity of hardwood would help the core resin to cure in time without lengthening the pressing period.

11.1.7 Series 3: Identifying appropriate configuration of hard/softwood particles in core and surfaces

The acidity of hardwood particles was identified as one of the critical factors affecting the quality of particleboard, as discussed in the findings of Series 2 of experiments. Since the pH variation among softwood and hardwood varies the board properties, it is essential to examine the configuration of hard/softwood within the particleboard.

- Six different hard/softwood configuration boards were made under the same processing conditions in order to investigate the effect of hard/softwood configuration on the properties of particleboard.
- Boards with S – H configuration were found to have optimal properties. This can be explained by the pH variation of hardwood and softwood particles. Hardwood with acidic characteristics cures faster at lower curing temperature when reacting with UF resin. Therefore, hardwood is more suitable for core layers, while softwood, which needs more time to cure, is better in the fast heating surfaces.
- Compared to hardwood particleboard and boards with mixtures of softwood and hardwood, particleboard from lower density softwood produces better properties. Lower density wood provides a high density compaction rate which leads to a higher contact surface between the particles. Therefore, particleboard made of low density wood is a more uniform product with a greater capacity to transmit loads between the particles, resulting in higher flexural and internal bonding properties.
- It is essential to understand the nature of wood materials for particleboard production as vast difference in the nature and amount of various chemical components within wood extracts can be found within a single species, depending on the age and growth site or the sampling location within the log.

11.1.8 Series 4: Particleboard production using S – H configuration

- Series 4 of experiments was designed with S – H configuration and four other variables. 16 boards were manufactured and tested. The S – H configuration of particleboard was validated as the most appropriate configuration to produce particleboard with optimal properties. Most of the boards as shown in Table 9.3 were found to obtain a significant bending and tensile strength complying with the standard of AS/NZS.

- Process models were generated for MOE, MOR, IB and density respectively. Comparison of the predicted values with actual experimental board properties verified that the models are sufficient to predict the particleboard properties within a certain range of variables. Errors found in each of the models were within acceptable range.

- These models were then used for board optimization. Optimization plots were generated from MINITAB to understand the factors affecting the predicted responses and allow modification of factor settings interactively. An optimal board recipe was obtained by adjusting the factor settings to match the desired properties. Optimized board was manufactured in the laboratory and tested for the required mechanical properties. This result further validates the models and confirms that they are adequate to be used in board optimization.

11.1.9 Surface studies and thermal analysis of hardwood and softwood composites

- GADDS was used to map the composites in order to check the uniformity of the samples on the surfaces. No significant differentiation between the hardwood and softwood board surfaces has been found in this study. However, previous research has been done to confirm the applicability of XRD for wood species. Therefore, further study is essential to advance the research.

- A better understanding of hardwood and softwood composition has been developed by using TGA. Decomposition rate with temperature increase was obtained. A regular

weight loss of hardwood and softwood composites can be found with increased temperature. Based on the outcomes of this study, hot pressing temperatures which will not adversely affect hardwood and softwood residues can be determined.

- The chemical analysis also confirmed that laboratory-manufactured boards have a similar cross-linking to those manufactured commercially, confirming the quality of the laboratory process.

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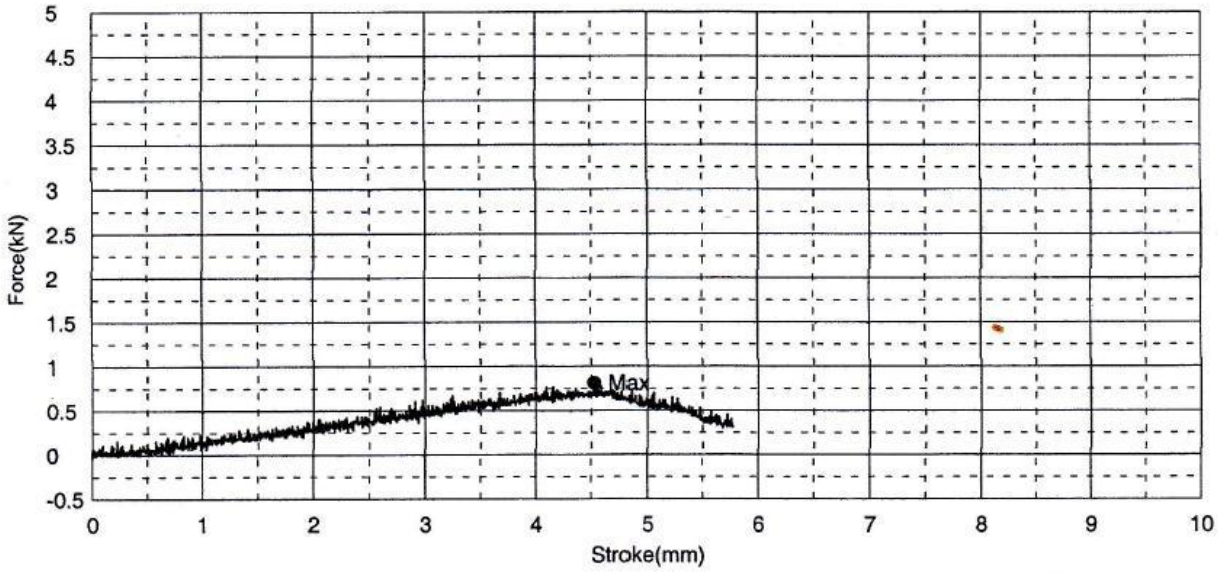
APPENDIX A: Three-point bending test results – Selected samples

Board N 6

Shape: Plate

	Thickness	Width	Lower Support
Units	mm	mm	mm
MOR	1.0000	1.0000	250.0000

Name	Max_Force
Units	kN
MOR	0.81250

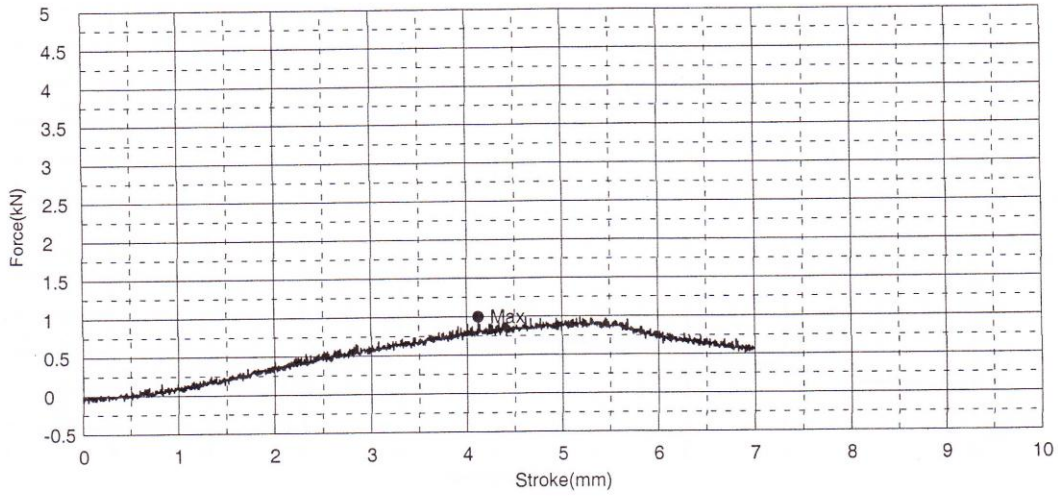


Board N 13

Shape: Plate

	Thickness	Width	Lower Support
Units	mm	mm	mm
MOR	1.0000	1.0000	250.0000

Name	Max_Force
Units	kN
MOR	1.00000



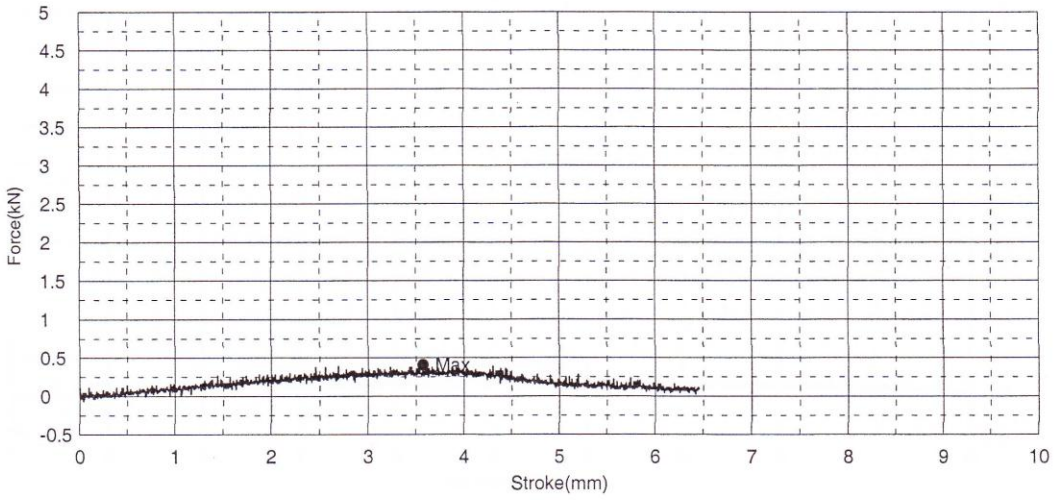
Comment

Board N 16

Shape: Plate

	Thickness	Width	Lower Support
Units	mm	mm	mm
MOR	1.0000	1.0000	250.0000

Name	Max_Force
Units	kN
MOR	0.40625



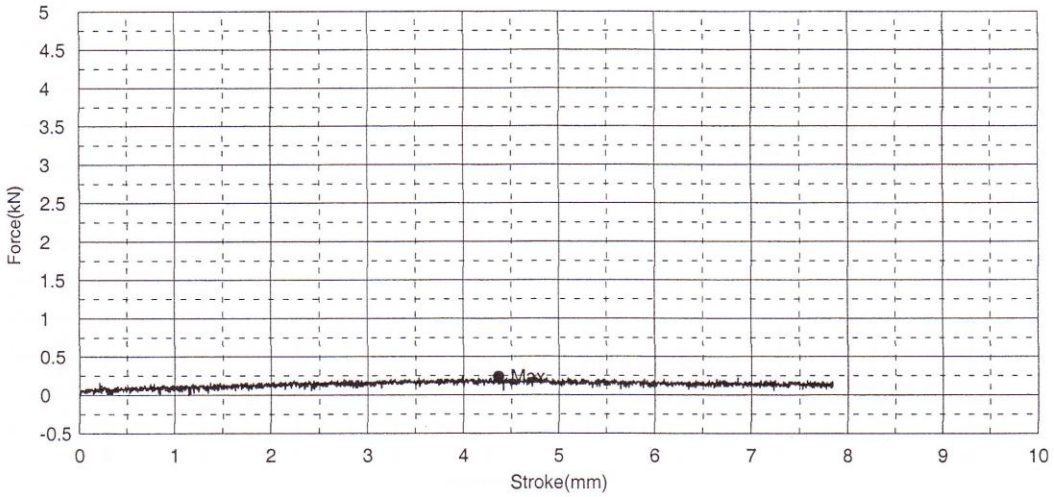
Comment

Board N 28

Shape: Plate

	Thickness	Width	Lower Support
Units	mm	mm	mm
MOR	1.0000	1.0000	250.0000

Name	Max_Force
Units	kN
MOR	0.23438



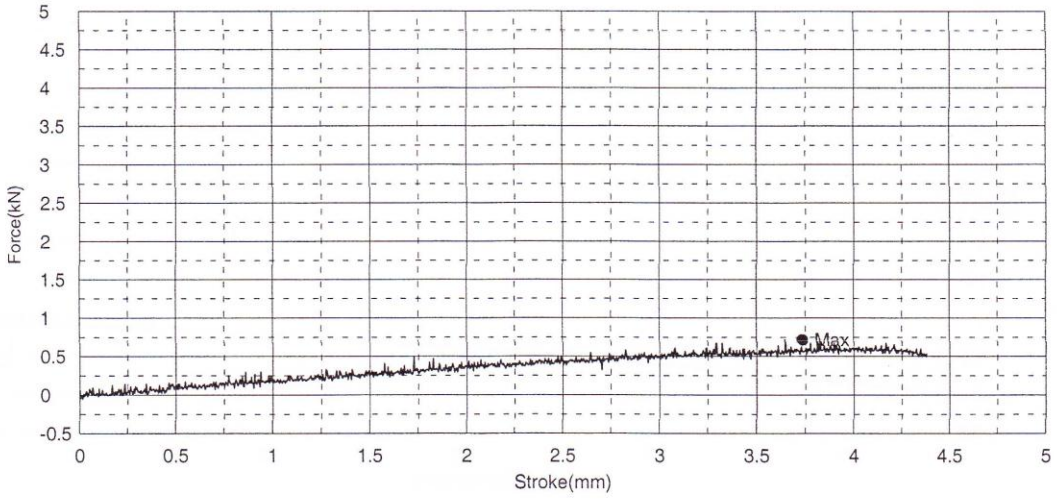
Comment

Board J

Shape: Plate

	Thickness	Width	Lower Support
Units	mm	mm	mm
MOR	1.0000	1.0000	250.0000

Name	Max_Force
Units	kN
MOR	0.71875



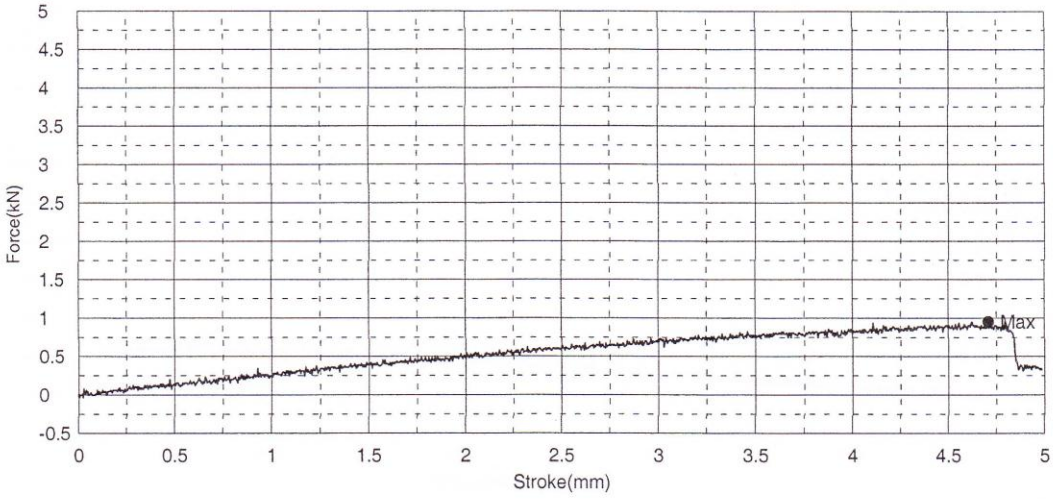
Comment

Board K

Shape: Plate

	Thickness	Width	Lower Support
Units	mm	mm	mm
MOR	1.0000	1.0000	250.0000

Name	Max_Force
Units	kN
MOR	0.95313



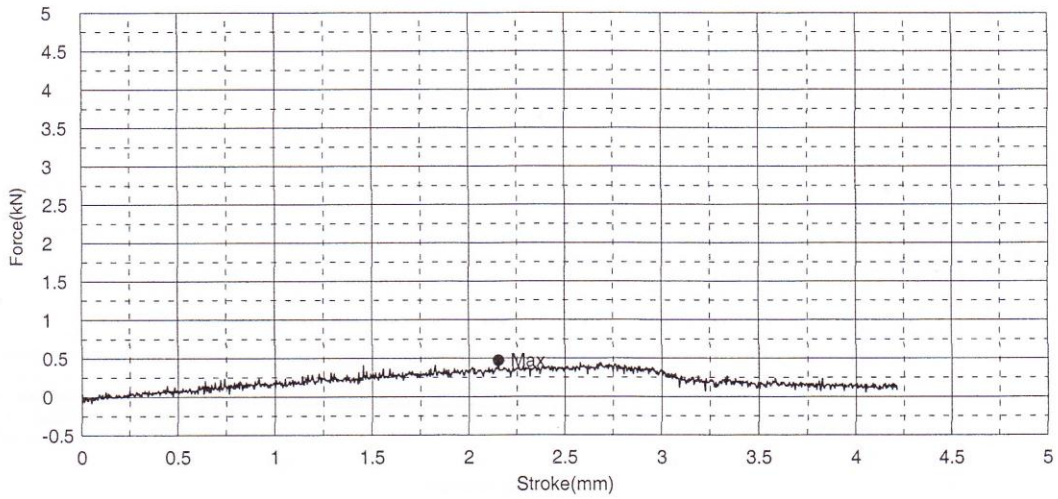
Comment

Board L

Shape: Plate

	Thickness	Width	Lower Support
Units	mm	mm	mm
MOR	1.0000	1.0000	250.0000

Name	Max_Force
Units	kN
MOR	0.46875



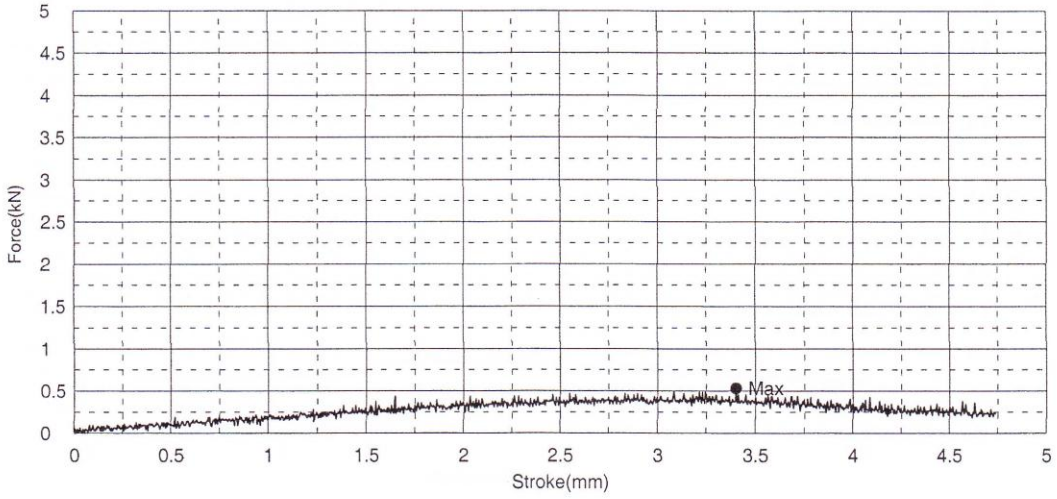
Comment

Board M

Shape: Plate

	Thickness	Width	Lower Support
Units	mm	mm	mm
MOR	1.0000	1.0000	250.0000

Name	Max_Force
Units	kN
MOR	0.53125



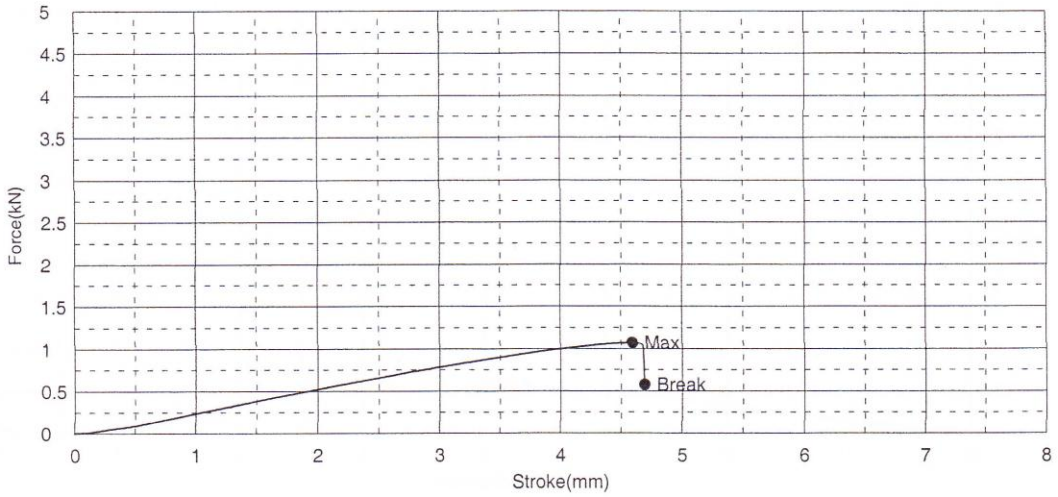
Comment

Board -Soft

Shape: Plate

	Thickness	Width	Lower Support
Units	mm	mm	mm
MOR	16.0000	100.0000	250.0000

Name	Max_Force	Break_Force
Units	kN	kN
MOR	1.07281	0.57563



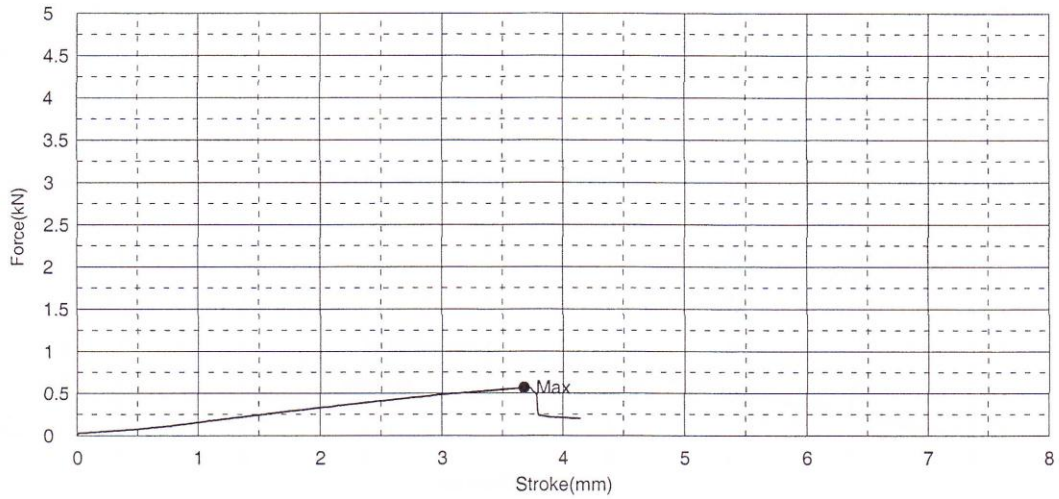
Comment

Board N hard

Shape: Plate

	Thickness	Width	Lower Support
Units	mm	mm	mm
MOR	16.0000	100.0000	250.0000

Name	Max_Force	Break_Force
Units	kN	kN
MOR	0.57094	-.-



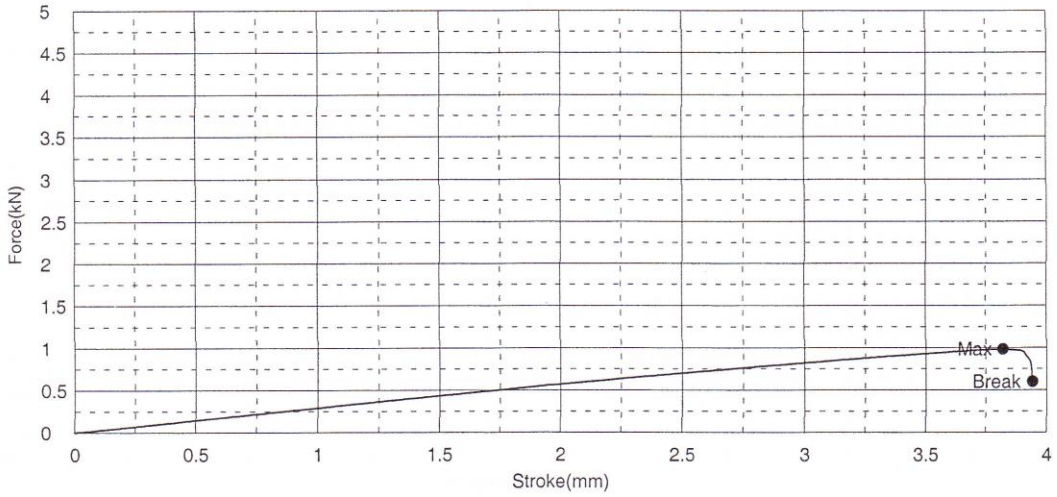
Comment

Board S-H

Shape: Plate

	Thickness	Width	Lower Support
Units	mm	mm	mm
MOR	16.0000	100.0000	250.0000

Name	Max_Force	Break_Force
Units	kN	kN
MOR	0.98375	0.59875



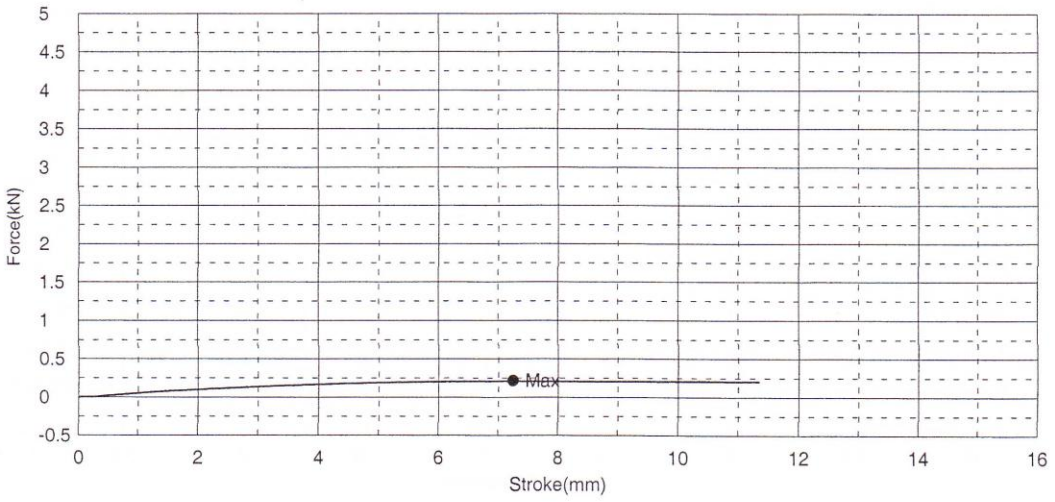
Comment

Board H-S

Shape: Plate

	Thickness	Width	Lower Support
Units	mm	mm	mm
MOR	16.0000	100.0000	250.0000

Name	Max_Force	Break_Force
Units	kN	kN
MOR	0.21625	-.-



Comment

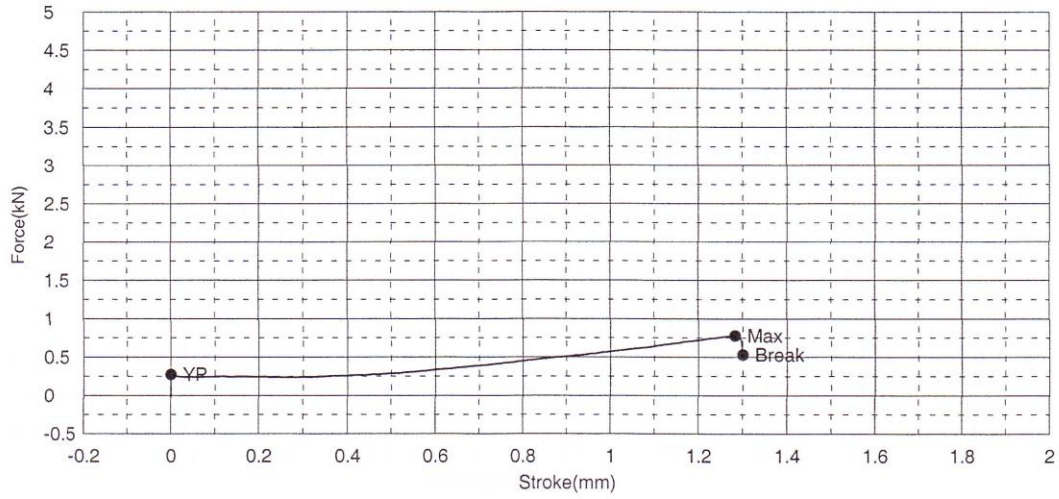
APPENDIX B: Tensile test results – Selected samples

tensile test N3

Shape: Rod

	Diameter	Gauge Length
Units	mm	mm
1 - 1	1.0000	100.0000

Name	YP_Force	Max_Force	Break_Force
Parameter	0.1 %/FS		
Units	kN	kN	kN
1 - 1	0.27312	0.77844	0.53062



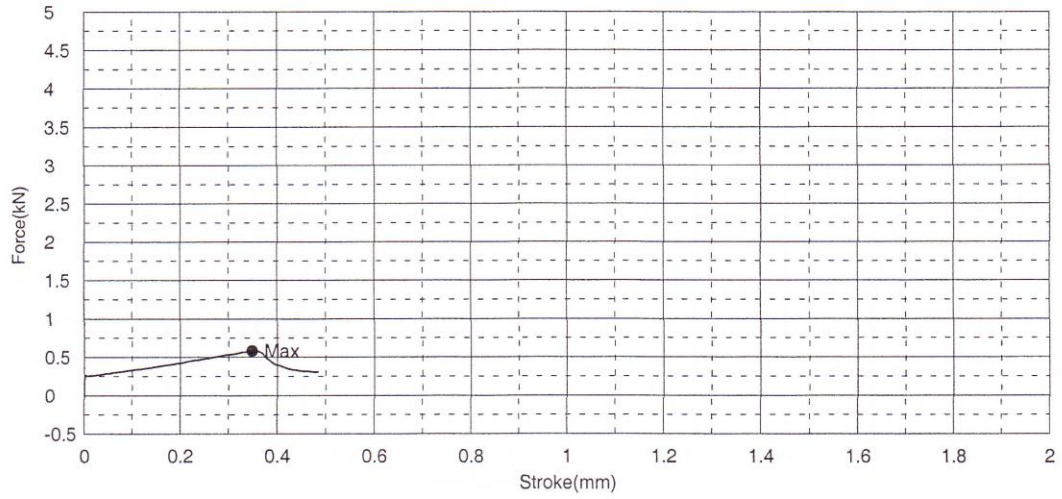
Comment

tensile test N4

Shape: Rod

	Diameter	Gauge Length
Units	mm	mm
1 - 1	1.0000	100.0000

Name	YP_Force	Max_Force	Break_Force
Parameter	0.1 %/FS		
Units	kN	kN	kN
1 - 1	-.-	0.58031	-.-



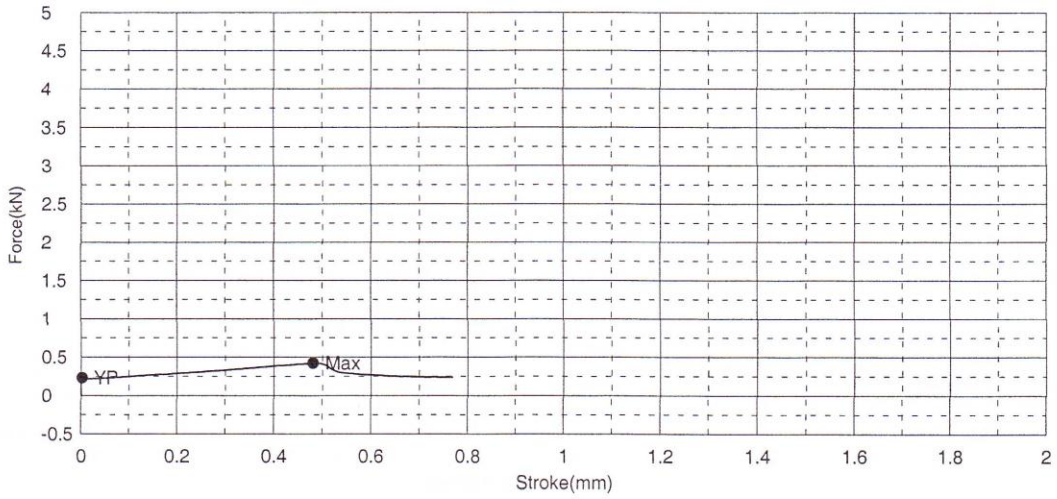
Comment

tensile test N9

Shape: Rod

	Diameter	Gauge Length
Units	mm	mm
1 - 1	1.0000	100.0000

Name	YP_Force	Max_Force	Break_Force
Parameter	0.1 %/FS		
Units	kN	kN	kN
1 - 1	0.23281	0.42188	-.-



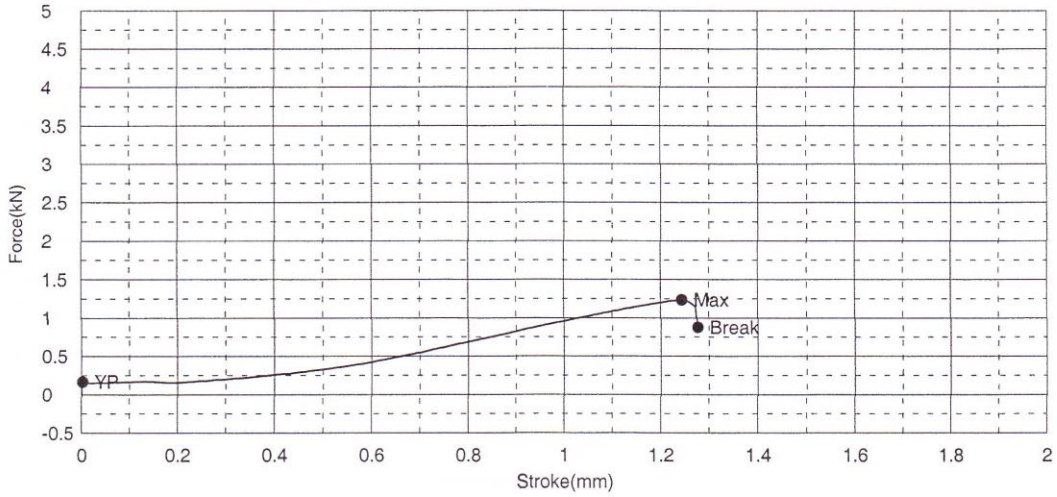
Comment

tensile test N30

Shape: Rod

	Diameter	Gauge Length
Units	mm	mm
1 - 1	1.0000	100.0000

Name	YP_Force	Max_Force	Break_Force
Parameter	0.1 %/FS		
Units	kN	kN	kN
1 - 1	0.16688	1.23625	0.87625



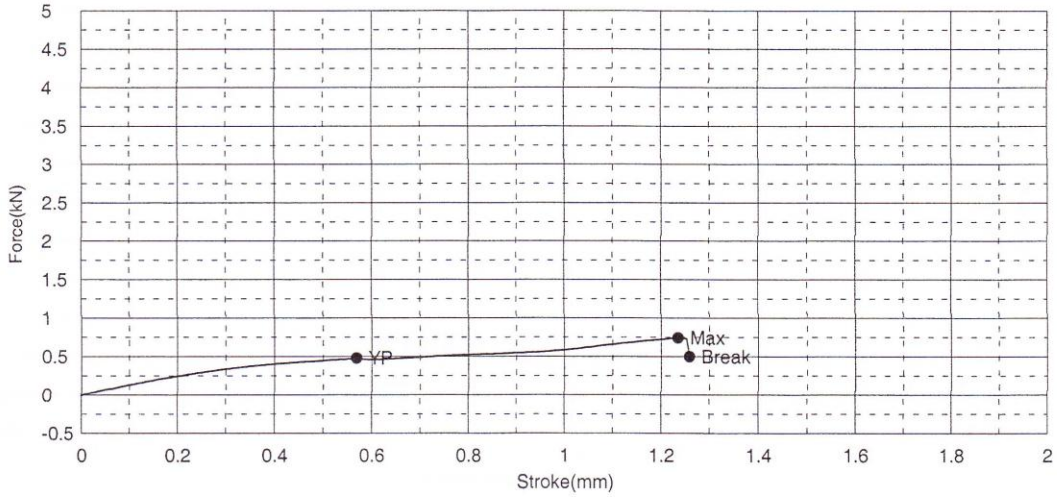
Comment

tensile test B

Shape: Rod

	Diameter	Gauge Length
Units	mm	mm
1 - 1	1.0000	100.0000

Name	YP_Force	Max_Force	Break_Force
Parameter	0.1 %/FS		
Units	kN	kN	kN
1 - 1	0.47938	0.74187	0.49875



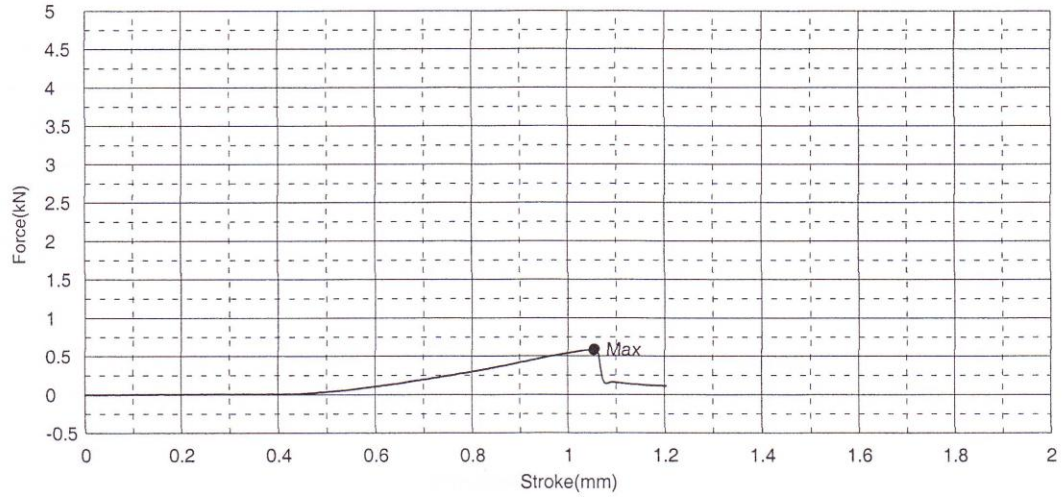
Comment

tensile test C

Shape: Rod

	Diameter	Gauge Length
Units	mm	mm
1 - 1	1.0000	100.0000

Name	YP_Force	Max_Force	Break_Force
Parameter	0.1 %/FS		
Units	kN	kN	kN
1 - 1	-.-	0.58875	-.-



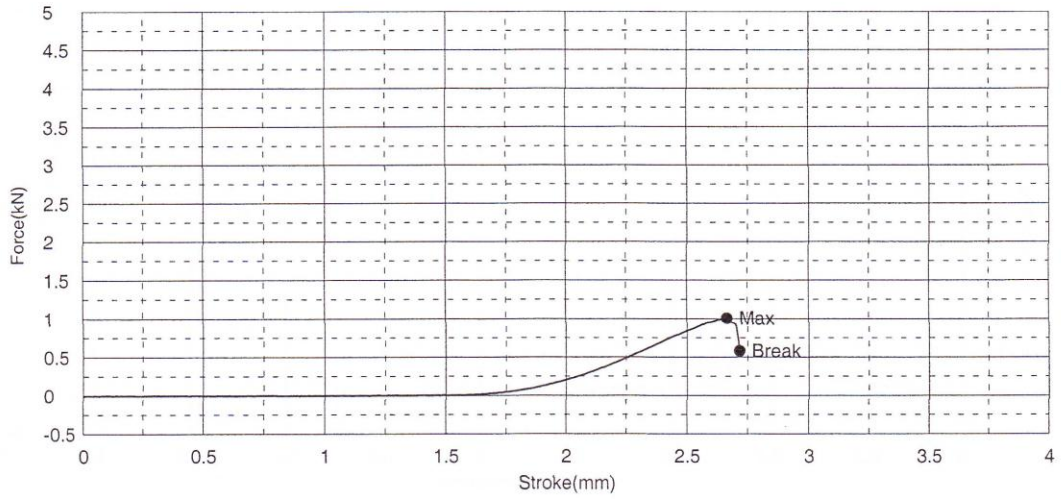
Comment

tensile test F

Shape: Rod

	Diameter	Gauge Length
Units	mm	mm
1 - 1	1.0000	100.0000

Name	YP_Force	Max_Force	Break_Force
Parameter	0.1 %/FS		
Units	kN	kN	kN
1 - 1	-.-	1.00875	0.58219



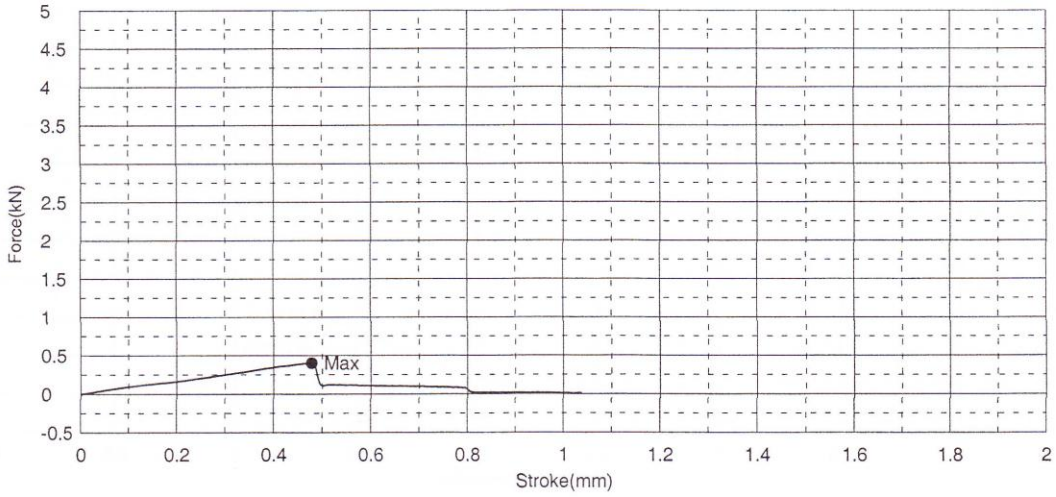
Comment

tensile test L

Shape: Rod

	Diameter	Gauge Length
Units	mm	mm
1 - 1	1.0000	100.0000

Name	YP_Force	Max_Force	Break_Force
Parameter	0.1 %/FS		
Units	kN	kN	kN
1 - 1	-.-	0.40156	-.-



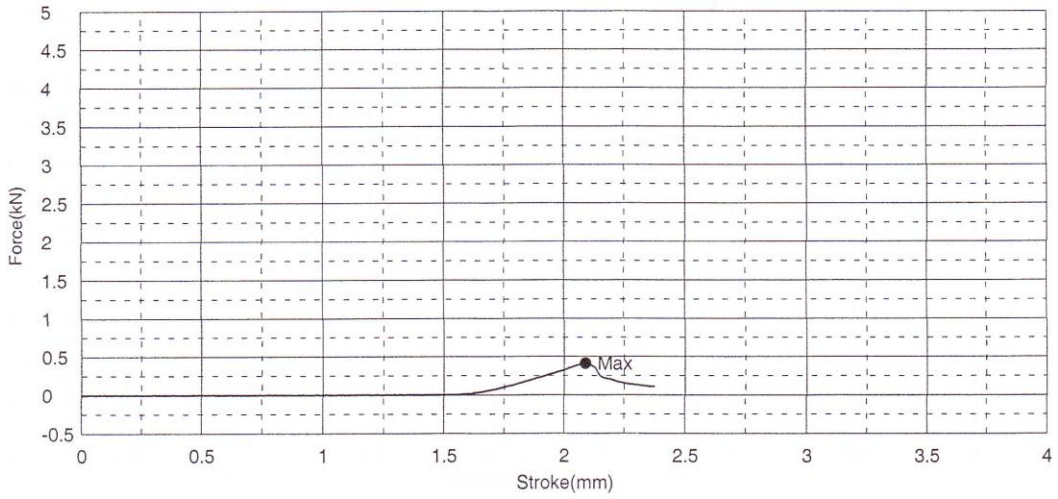
Comment

tensile test Hard

Shape: Rod

	Diameter	Gauge Length
Units	mm	mm
1 - 1	1.0000	100.0000

Name	YP_Force	Max_Force	Break_Force
Parameter	0.1 %/FS		
Units	kN	kN	kN
1 - 1	-.-	0.41125	-.-



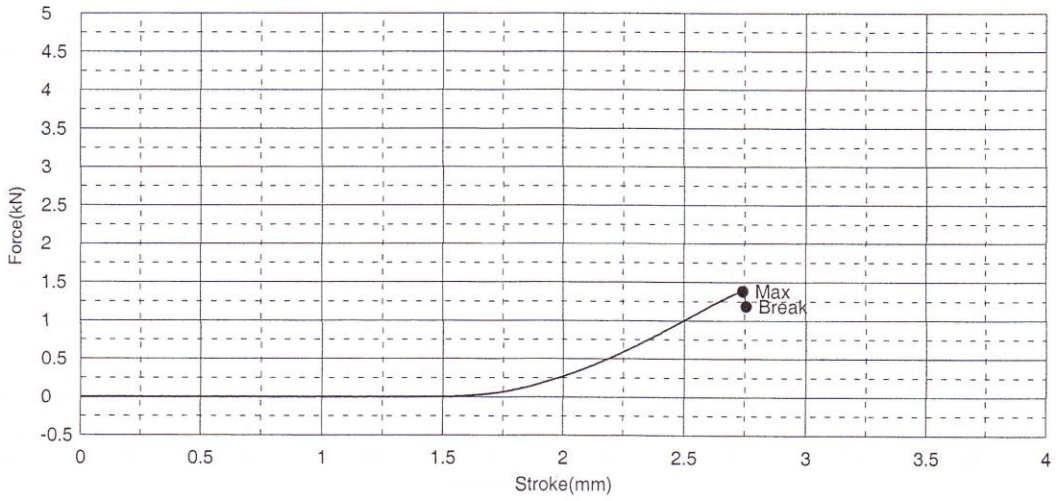
Comment

tensile test Soft

Shape: Rod

	Diameter	Gauge Length
Units	mm	mm
1 - 1	1.0000	100.0000

Name	YP_Force	Max_Force	Break_Force
Parameter	0.1 %/FS		
Units	kN	kN	kN
1 - 1	-.-	1.38312	1.19031



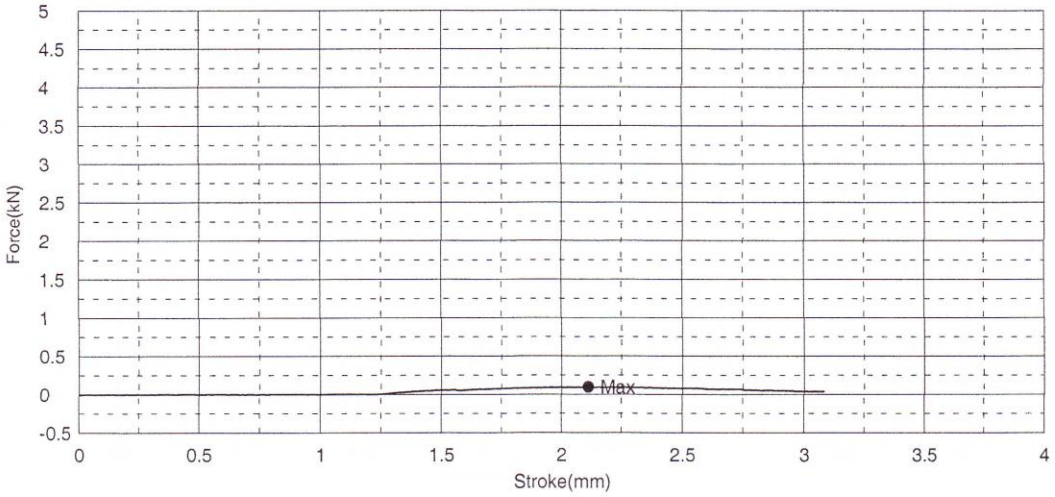
Comment

tensile test H-S

Shape: Rod

	Diameter	Gauge Length
Units	mm	mm
1 - 1	1.0000	100.0000

Name	YP_Force	Max_Force	Break_Force
Parameter	0.1 %/FS		
Units	kN	kN	kN
1 - 1	-.-	0.09469	-.-



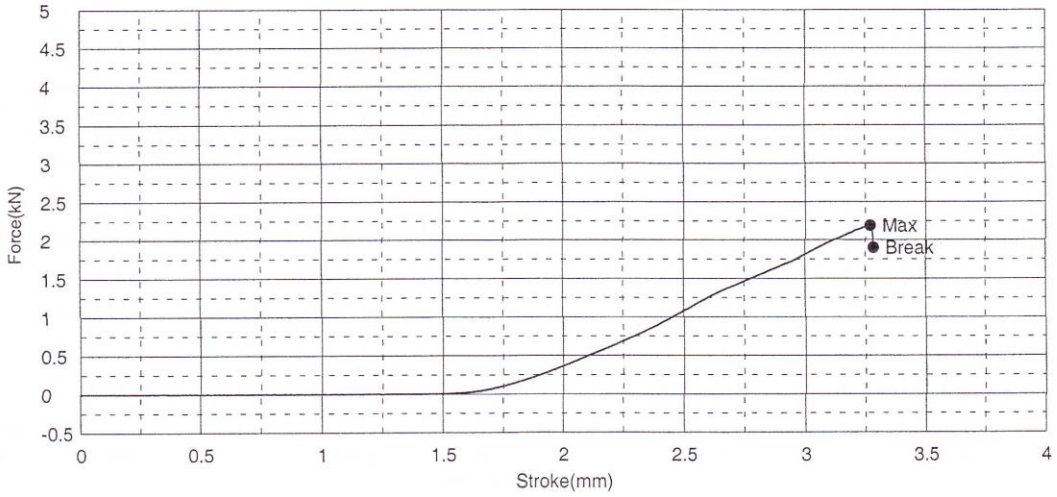
Comment

tensile test S-H

Shape: Rod

	Diameter	Gauge Length
Units	mm	mm
1 - 1	1.0000	100.0000

Name	YP_Force	Max_Force	Break_Force
Parameter	0.1 %/FS		
Units	kN	kN	kN
1 - 1	-.-	2.19219	1.90000



Comment

APPENDIX C: Product information sheet for UF resin

WOODCHEM T0076

Section 1 - CHEMICAL PRODUCT AND COMPANY IDENTIFICATION

PRODUCT NAME
WOODCHEM T0076

SYNONYMS

PRODUCT USE
Used in composite board. Cured by heating.

SUPPLIER

Company: Woodchem Australia P/L
Address:
Locked Bag 6
Oberon
NSW, 2787
AUS

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Section 2 - HAZARDS IDENTIFICATION

STATEMENT OF HAZARDOUS NATURE
HAZARDOUS SUBSTANCE. NON-DANGEROUS GOODS. According to the Criteria of NOHSC, and the ADG Code.

POISONS SCHEDULE
None

RISK
Irritating to eyes and skin.
May cause SENSITISATION by skin contact.

SAFETY
Do not breathe gas/fumes/vapour/spray.
Avoid contact with skin.
Wear eye/face protection.
To clean the floor and all objects contaminated by this material, use water.
In case of contact with eyes, rinse with plenty of water and contact Doctor or Poisons Information Centre.
If swallowed, IMMEDIATELY contact Doctor or Poisons Information Centre. (show this container or label).

Section 3 - COMPOSITION / INFORMATION ON INGREDIENTS

NAME	CAS RN	%
urea/ formaldehyde resin	9011-05-6	>60
formaldehyde.	50-00-0	0.5
water	7732-18-5	<40

continued...

NOTE: Manufacturer has supplied full ingredient information to allow CHEMWATCH assessment.

Section 4 - FIRST AID MEASURES

SWALLOWED

- If swallowed do NOT induce vomiting.
- If vomiting occurs, lean patient forward or place on left side (head-down position, if possible) to maintain open airway and prevent aspiration.
- Observe the patient carefully.
- Never give liquid to a person showing signs of being sleepy or with reduced awareness; i.e. becoming unconscious.
- Give water to rinse out mouth, then provide liquid slowly and as much as casualty can comfortably drink.
- Seek medical advice.

EYE

If this product comes in contact with the eyes:

- Wash out immediately with fresh running water.
- Ensure complete irrigation of the eye by keeping eyelids apart and away from eye and moving the eyelids by occasionally lifting the upper and lower lids.
- If pain persists or recurs seek medical attention.
- Removal of contact lenses after an eye injury should only be undertaken by skilled personnel.

SKIN

If skin contact occurs:

- Immediately remove all contaminated clothing, including footwear
- Flush skin and hair with running water (and soap if available).
- Seek medical attention in event of irritation.

INHALED

- If fumes or combustion products are inhaled remove from contaminated area.
- Lay patient down. Keep warm and rested.
- Prostheses such as false teeth, which may block airway, should be removed, where possible, prior to initiating first aid procedures.
- Apply artificial respiration if not breathing, preferably with a demand valve resuscitator, bag-valve mask device, or pocket mask as trained. Perform CPR if necessary.
- Transport to hospital, or doctor.

NOTES TO PHYSICIAN

Treat symptomatically.

Section 5 - FIRE FIGHTING MEASURES

EXTINGUISHING MEDIA

- There is no restriction on the type of extinguisher which may be used.

FIRE FIGHTING

- Alert Fire Brigade and tell them location and nature of hazard.
- Wear breathing apparatus plus protective gloves for fire only.

continued...

- Prevent, by any means available, spillage from entering drains or water courses.
- Use fire fighting procedures suitable for surrounding area.
- DO NOT approach containers suspected to be hot.
- Cool fire exposed containers with water spray from a protected location.
- If safe to do so, remove containers from path of fire.
- Equipment should be thoroughly decontaminated after use.

FIRE/EXPLOSION HAZARD
- The material is not readily combustible under normal conditions.
- However, it will break down under fire conditions and the organic component may burn.
- Not considered to be a significant fire risk.
- Heat may cause expansion or decomposition with violent rupture of containers.
- Decomposes on heating and may produce toxic fumes of carbon monoxide (CO).
- May emit acrid smoke.

Other decomposition products include carbon dioxide (CO_2), nitrogen oxides (NO_x) and aldehydes.

FIRE INCOMPATIBILITY
Avoid contamination with strong oxidising agents as ignition may result.

HAZCHEM
None

Personal Protective Equipment
PERSONAL PROTECTION EQUIPMENT
Gas tight chemical resistant suit.
Limit exposure duration to 1 BA set - 30 mins.

Section 6 - ACCIDENTAL RELEASE MEASURES

EMERGENCY PROCEDURES

MINOR SPILLS
Slippery when spilt.
- Clean up all spills immediately.
- Avoid breathing vapours and contact with skin and eyes.
- Control personal contact by using protective equipment.
- Contain and absorb spill with sand, earth, inert material or vermiculite.
- Wipe up.
- Place in a suitable labelled container for waste disposal.

MAJOR SPILLS
Slippery when spilt.
Minor hazard.
- Clear area of personnel.
- Alert Fire Brigade and tell them location and nature of hazard.
- Control personal contact by using protective equipment as required.
- Prevent spillage from entering drains or water ways.
- Contain spill with sand, earth or vermiculite.
- Collect recoverable product into labelled containers for recycling.
- Absorb remaining product with sand, earth or vermiculite and place in appropriate containers for disposal.

continued...

- Wash area and prevent runoff into drains or waterways.
- If contamination of drains or waterways occurs, advise emergency services.

Personal Protective Equipment advice is contained in Section 8 of the MSDS.

Section 7 - HANDLING AND STORAGE

PROCEDURE FOR HANDLING

- Limit all unnecessary personal contact.
- Wear protective clothing when risk of exposure occurs.
- Use in a well-ventilated area.
- Avoid contact with incompatible materials.
- When handling, DO NOT eat, drink or smoke.
- Keep containers securely sealed when not in use.
- Avoid physical damage to containers.
- Always wash hands with soap and water after handling.
- Work clothes should be laundered separately.
- Use good occupational work practice.
- Observe manufacturer's storing and handling recommendations.
- Atmosphere should be regularly checked against established exposure standards to ensure safe working conditions are maintained.

SUITABLE CONTAINER

- Lined metal can, Lined metal pail/ can
- Plastic pail
- Polyliner drum
- Packing as recommended by manufacturer.
- Check all containers are clearly labelled and free from leaks.

STORAGE INCOMPATIBILITY

Avoid storage with oxidisers and acids.

STORAGE REQUIREMENTS

- Store in original containers.
- Keep containers securely sealed.
- Store in a cool, dry, well ventilated area.
- DO NOT allow to freeze.
- Store away from incompatible materials.
- Protect containers against physical damage and check regularly for leaks.
- Observe manufacturer's storing and handling recommendations.

Store below 30 deg. C.

Section 8 - EXPOSURE CONTROLS / PERSONAL PROTECTION

EXPOSURE CONTROLS

Source	Material	TWA ppm	TWA mg/m³	STEL ppm	STEL mg/m³	Peak ppm	Peak mg/m³
Australian Exposure Standards	Formaldehyde	1	1.2	2	2.5		

No data available for urea/ formaldehyde resin as (CAS: 9011-05-6) / (CAS: 39327-95-2) / (CAS: 56779-89-6) / (CAS: 57608-68-1) / (CAS: 57657-45-
No data available for water as (CAS: 7732-18-5)

continued...

None assigned. Refer to individual constituents.

EXPOSURE STANDARDS FOR MIXTURE

"Worst Case" computer-aided prediction of spray/ mist or fume/ dust components and concentration:

"Worst Case" computer-aided prediction of spray/ mist or fume/ dust components and concentration:
Composite Exposure Standard for Mixture (TWA) :100 mg/m³.

INGREDIENT DATA

UREA/ FORMALDEHYDE RESIN:
Dusts not otherwise classified, as inspirable dust;
ES TWA: 10 mg/m³.

FORMALDEHYDE.:
Odour Threshold Value: 0.98 ppm (recognition)
NOTE: Detector tubes for formaldehyde, measuring in excess of 0.2 ppm are available commercially.
Formaldehyde vapour exposure:
Primary irritation is dependent on duration of exposure and individual susceptibility.
The following are typical symptoms encountered at various exposure levels.
0.1 ppm - Lower level of mucous eye, nose and throat irritation
0.8 ppm - Typical threshold of perception
1-2 ppm - Typical threshold of irritation
2-3 ppm - Irritation of eyes, nose and throat

WATER:
No exposure limits set by NOHSC or ACGIH.

PERSONAL PROTECTION

EYE
- Safety glasses with side shields; or as required,
- Chemical goggles.
- Contact lenses may pose a special hazard; soft contact lenses may absorb and concentrate irritants. A written policy document, describing the wearing of lens or restrictions on use, should be created for each workplace or task. This should include a review of lens absorption and adsorption for the class of chemicals in use and an account of injury experience. Medical and first-aid personnel should be trained in their removal and suitable equipment should be readily available. In the event of chemical exposure, begin eye irrigation immediately and remove contact lens as soon as practicable. Lens should be removed at the first signs of eye redness or irritation - lens should be removed in a clean environment only after workers have washed hands thoroughly. [CDC NIOSH Current Intelligence Bulletin 59].

HANDS/FEET
Neoprene gloves. Safety footwear.

OTHER
- Overalls.
- Eyewash unit.

continued...

RESPIRATOR

Selection of the Class and Type of respirator will depend upon the level of breathing zone contaminant and the chemical nature of the contaminant. Protection Factors (defined as the ratio of contaminant outside and inside the mask) may also be important.

Breathing Zone Level ppm (volume)	Maximum Protection Factor	Half-face Respirator	Full-Face Respirator
1000	10	BAX-AUS P	-
1000	50	-	BAX-AUS P
5000	50	Airline *	-
5000	100	-	BAX-2 P
10000	100	-	BAX-3 P
	100+		Airline**

* - Continuous Flow ** - Continuous-flow or positive pressure demand.

The local concentration of material, quantity and conditions of use determine the type of personal protective equipment required. For further information consult site specific CHEMWATCH data (if available), or you

ENGINEERING CONTROLS

General exhaust is adequate under normal operating conditions. Local exhaust ventilation may be required in specific circumstances. If risk of overexposure exists, wear approved respirator. Correct fit is essential to obtain adequate protection. Provide adequate ventilation in warehouse or closed storage areas.

Section 9 - PHYSICAL AND CHEMICAL PROPERTIES

APPEARANCE

White/opaque liquid with a mild formaldehyde odour; mixes with water.

PHYSICAL PROPERTIES

Liquid.
Mixes with water.

Molecular Weight: Not applicable
Melting Range (°C): Not available
Solubility in water (g/L): Miscible
pH (1% solution): Not available
Volatile Component (%vol): Not available
Relative Vapour Density (air=1): Not available
Lower Explosive Limit (%): Not applicable
Autoignition Temp (°C): Not applicable
State: Liquid

Boiling Range (°C): Not available
Specific Gravity (water=1): 1.29 approx.
pH (as supplied): 7.8 approx.
Vapour Pressure (kPa): Not available
Evaporation Rate: Not available
Flash Point (°C): Not applicable
Upper Explosive Limit (%): Not applicable
Decomposition Temp (°C): Not available

Section 10 - CHEMICAL STABILITY AND REACTIVITY INFORMATION

CONDITIONS CONTRIBUTING TO INSTABILITY

- Presence of incompatible materials.
- Product is considered stable.
- Hazardous polymerisation will not occur.

continued...

Section 11 - TOXICOLOGICAL INFORMATION

POTENTIAL HEALTH EFFECTS

ACUTE HEALTH EFFECTS

SWALLOWED
The liquid is discomforting to the gastro-intestinal tract if swallowed.
Ingestion may result in nausea, abdominal irritation, pain and vomiting.
Considered an unlikely route of entry in commercial/industrial environments.

EYE
The liquid is highly discomforting to the eyes.
The vapour is discomforting to the eyes.

SKIN
The liquid is slightly discomforting to the skin if exposure is prolonged and is capable of causing skin sensitisation and allergic skin reactions which may lead to dermatitis.
Sensitisation may result in allergic dermatitis responses including rash, itching, hives or swelling of extremities.

INHALED
The vapour is discomforting to the upper respiratory tract.
Inhalation hazard is increased at higher temperatures.

CHRONIC HEALTH EFFECTS
Principal routes of exposure are usually by skin contact / eye contact and inhalation of vapour. The material contains residues of free formaldehyde which may irritate the eyes or mucous membranes and produce an allergic response if used in confined spaces or heated.

TOXICITY AND IRRITATION
Not available. Refer to individual constituents.
unless otherwise specified data extracted from RTECS - Register of Toxic Effects of Chemical Substances

UREA/ FORMALDEHYDE RESIN:

TOXICITY	IRRITATION
Oral (rat) LD50: 8394 mg/kg	Skin (rabbit): 500 mg/24h-SEVERE
Inhalation (rat) LC50: >167 mg/m3/4h	Eye (rabbit): 0.1 ul/24h -SEVERE
Dermal (rat) LD50: >2100 mg/kg	
Oral (mouse) LD50: 6361 mg/kg	

Somnolence, impaired liver function tests, changes in leucocyte (WBC) count recorded.
NOTE: Substance has been shown to be mutagenic in at least one assay, or belongs to a family of chemicals producing damage or change to cellular DNA.

FORMALDEHYDE.:

TOXICITY	IRRITATION
Oral (woman) LDLo: 108 mg/kg	Skin (human): 0.15 mg/3d-I Mild
Oral (man) TDLo: 643 mg/kg	Skin (rabbit): 2 mg/24H SEVERE
Oral (rat) LD50: 100 mg/kg	Eye (human): 4 ppm/5m
Inhalation (man) TCLo: 0.3 mg/m3	Eye (rabbit): 0.75 mg/24H SEVERE
Inhalation (rat) LC50: 203 mg/m3	

continued...

Dermal (rabbit) LD50: 270 mg/kg
WARNING: This substance has been classified by the IARC as Group 1: CARCINOGENIC TO HUMANS.
Tenth Annual Report on Carcinogens: Substance anticipated to be Carcinogen
[National Toxicology Program: U.S. Dep. of Health & Human Services 2002].

WATER:
No significant acute toxicological data identified in literature search.

Section 12 - ECOLOGICAL INFORMATION

Refer to data for ingredients, which follows:

UREA/ FORMALDEHYDE RESIN:
Slowly, but not readily biodegradable.
TOC - removal 28 days; 61% BOD28; 0.62 mg/mg substance.
Nitrification 28 days; 0.45 mg NO3-N/mg substance.

FORMALDEHYDE.:
The material is classified as an ecotoxin* because the Fish LC50 (96 hours) is less than or equal to 0.1 mg/l

* Classification of Substances as Ecotoxic (Dangerous to the Environment)
Appendix 8, Table 1
Compiler's Guide for the Preparation of International Chemical Safety Cards:
1993 Commission of the European Communities.

Section 13 - DISPOSAL CONSIDERATIONS

- Recycle wherever possible or consult manufacturer for recycling options.
- Consult State Land Waste Management Authority for disposal.
- Bury residue in an authorised landfill.
- Recycle containers if possible, or dispose of in an authorised landfill.

Section 14 - TRANSPORTATION INFORMATION

Dangerous Goods Class: None
Subrisk: None
UN/NA Number: None
Packing Group: None
Labels Required:
Additional Shipping Information:
International Transport Regulations:
IMO Dangerous Goods class: None
IMO Packing group: None
IATA Dangerous goods class: None
Cargo Instructions:
Cargo Max:
Passenger Instructions:
Passenger Max:
Special Provisions: None, None

continued...

HAZCHEM

None

Section 15 - REGULATORY INFORMATION

POISONS SCHEDULE

None

REGULATIONS

urea/ formaldehyde resin (CAS: 9011-05-6) is found on the following regulatory lists:
Australian Inventory of Chemical Substances (AICS)

formaldehyde. (CAS: 50-00-0) is found on the following regulatory lists:
Australian Inventory of Chemical Substances (AICS)
Australian Poisons Schedule
Australia High Volume Industrial Chemical List (HVICL)

water (CAS: 7732-18-5) is found on the following regulatory lists:
Australian Inventory of Chemical Substances (AICS)

No data available for urea/ formaldehyde resin as CAS: 39327-95-2, CAS: 56779-89-6, CAS: 57608-68-1, CAS: 57657-45-1, CAS: 57762-61-5, CAS: 60267-46-1, CAS: 60831-80-3.

Section 16 - OTHER INFORMATION

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